## Quality Assurance Manual

Prepared for the use of: A & B Environmental Services, Inc 10100 East Freeway, Suite 100 Houston, Texas 77029

Revision February, 2006

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## Table of Contents

| <u>Section</u> | <u>Topic</u>                            | <u>Page</u> |
|----------------|---|-------------|
| 1              | Introduction                            | 1           |
| 2              | Quality Assurance Policy                | 2           |
| 3              | Scope of Document                       | 2 5         |
| 4              | Definition of Terms                     | 7           |
| 5              | Personnel Qualifications and Training   | 8           |
|                | Personnel Qualifications                | 8           |
|                | Training                                | 10          |
| 6              | Responsibilities and Authorities        | 12          |
|                | Organizational and Management Structure | 17          |
|                | Interdepartmental Communication         | 17          |
| 7              | Equipment                               | 18          |
| 8              | Sampling Procedures                     | 21          |
|                | Sampling Program Operations             | 22          |
|                | Sample Containers                       | 22          |
|                | Holding Times                           | 22          |
|                | Sub-Samples                             | 23          |
| 9              | Sample Custody                          | 24          |
|                | Receipt                                 | 24          |
|                | Acceptance                              | 24          |
|                | Sample Rejection                        | 24          |
|                | Storage                                 | 25          |
|                | Sample Disposal                         | 26          |
| 10             | Calibration Procedures and Frequency    | 30          |
|                | Instrument Calibration and Tuning       | 30          |
|                | Calibration Records                     | 30          |
| 11             | Analytical Procedures                   | 36          |
|                | Adoption                                | 36          |
|                | SOP                                     | 36          |
|                | Demonstration of Performance            | 37          |
|                | Reviewing and Revising                  | 38          |
| 12             | Internal QC Checks                      | 39          |
|                | Quality of Test Results                 | 39          |
|                | Blanks                                  | 39          |
|                | Duplicates                              | 40          |
|                | Laboratory Control Samples              | 41          |

ab-q010-0705 i

## A & B Labs QA Plan

| <u>Section</u>              | <u>Topic</u>   | <u>Page</u> |
|-----------------------------|--|-------------|
|                             | Matrix Spikes and Matrix Spike Duplicates  | 41          |
|                             | Surrogate Recoveries   | 42          |
|                             | Proficiency Testing  | 42          |
| 13                          | Routine Procedures Used to Assess Data Quality   |             |
|                             | Data Quality Assessment  | 44          |
|                             | Reporting Limits   | 47          |
| 14                          | Data Reduction, Validation and Reporting   | 48          |
|                             | Data Reduction and Validation  | 48          |
|                             | Analytical reporting   | 50          |
|                             | Client Confidentiality   | 51          |
| 15                          | Performance and System Audits  | 53          |
| 16                          | Nonconformance and Corrective Action   |             |
|                             | Nonconformance   | 55          |
|                             | Root Cause Investigation   | 56          |
|                             | Corrective Action  | 58          |
|                             | Preventative Action  | 58          |
| 17                          | Client Communications  | 61          |
| 18                          | QA Reports to Management   | 63          |
| 19                          | Laboratory Documentation   | 64          |
|                             | Records  | 64          |
|                             | Field Records  | 64          |
|                             | Laboratory Notebooks   | 64          |
|                             | Control Charts   | 65          |
|                             | $SOP_S$  | 65          |
|                             | Project Files  | 66          |
|                             | Computer Records   | 66          |
|                             | Laboratory Documents and Forms   | 66          |
| 20                          | Laboratory Consumables   | 70          |
| 21                          | Preventive Maintenance   | 72          |
| 22                          | Advertising  | 73          |
| 23                          | References   | 74          |
| Appendix I                  | Organizational Chart   |             |
| Appendix II                 | Required Containers, Preservation Techniques, and Holding Times For Solid Waste and Wastewater |             |
| Appendix III<br>Appendix IV | Format For Analytical Standard Operating Procedures (SOPs)<br>Analyst Training Checklist       |             |
| Appendix V                  | Bench Level Data Review Checklist  |             |
| Appendix VI                 | Test Methods used by $A\mathcal{C}B$   |             |

ab-q010-0705 ii

## **List of Figures**

| Figure |   | Page |
|--------|---|------|
| 9-1    | Chain of Custody Record                             | 27   |
| 9-2    | Sample Collection, Transport, and Holding Flowchart | 28   |
| 9-3    | Sample Condition Checklist                          | 29   |
| 14-1   | Data Validation Process                             | 52   |
| 16-1   | Work flow   | 56   |
| 16-2   | Corrective Action Report                            | 60   |

ab-q010-0705 iii

## 1. INTRODUCTION

A & B Labs is a minority owned laboratory formed in 1988 to provide quality service to the Environmental and Industrial Hygiene Industry. A & B is an independent, commercial testing laboratory that provides services to varied industrial clients and to governmental agencies. A & B's technical staff has the experience and knowledge in understanding regulatory limits and permit requirements, which is essential in helping clients meet their project goals. A & B strives to meet or exceed all applicable regulatory accreditation requirements.

### 2. QUALITY ASSURANCE POLICY

A & B Labs is committed to providing quality analytical services to our clients. A & B depends not only on the skills, abilities, and commitments of its employees but also on their integrity and common sense. A & B employees participate in relationships that involve, clients, suppliers, fellow employees, and the communities of which we are a part. The following serve as a basis for shaping these relationships.

We will strive to increase customer satisfaction by providing quality services that are responsive to their requirements.

We will treat our suppliers fairly, honestly, and objectively.

We will treat fellow employees in a fair and even-handed manner and foster a culture rich in diversity that is based on trust, mutual respect, teamwork, and integrity.

We will pursue growth and profitability objectives, while keeping ethical standards in the forefront of our activities.

In the communities of which we are members, we will act ethically and as responsible citizens in compliance with the law.

The management of A & B seeks to avoid any activities that would diminish confidence in its competence, impartiality, judgment, and operational integrity by:

Encouraging employees to seek guidance when needed. The President, Laboratory Director, or supervisor will listen, discuss, and make decisions on any concern brought to them either in person or in writing. These concerns may be handled on a one to one basis or brought before the management committee.

Asking employees to report to an officer of the company any suspected violations of our standards or policy. The president, director, or any officer reported to, will treat all reports confidentially to the extent permitted under law.

Fostering an atmosphere of co-operation between employees and between the

company and clients. Lab wide meetings are routinely held to discuss safety, QA, problems, and plans. Workload is shared when necessary. With notice, clients are welcome to tour the lab. Customer Service is responsive to customer inquiries and complaints. Customer complaints are recorded and acted on in a timely fashion.

Providing accurate data based on accepted analytical procedures in a timely fashion. Analysts are trained in and knowledgeable about the methods they perform. Continuing education and professional development is encouraged and provided to employees.

Seeking to employ measures that will ensure absence of prejudice with respect to race, creed, gender, and nationality. All issues are heard with an unbiased mind and discussed before resolution is determined.

To ensure the production of scientifically sound, legally defensible data of known, documentable and verifiable quality, an extensive Quality Assurance (QA) program has been implemented. This program relies on well-documented procedures, a comprehensive audit system, and management support, for its effectiveness.

This Quality Assurance manual has been developed to codify procedures that ensure that quality data is provided to our clients. This manual is intended to provide methods for oversight for many of the procedures and routines followed in the lab and to ensure that the highest achievable standards are met. Standard Operating Procedures (SOPs) have been prepared for analytical methods and operations used by A & B and are the final reference for laboratory procedures.

Equipment and supplies shall be obtained as needed to provide for the utilization of established technology and methodology. All of A & B's laboratory personnel shall be fully qualified and trained to perform assigned analyses. The performance of all laboratory personnel and equipment shall be continuously monitored and documented. Corrective action must be implemented and documented immediately if a quality problem arises.

It is the intention of A & B Labs that no analyst be subjected to undue pressure which could compromise the quality of their work. Possible sources of pressure might be internal (management or deadline), or external (client complaints or priority requests). If any analyst feels pressured from within the company, or outside the company by client requests, the QA Officer, Technical Manager, Department Manager, Project Manager,

or Customer Service Officer should be informed immediately. These Officers will resolve the problem. This may include such measures as assigning more people to the work load or notifying the customer of company policy.

In order to minimize pressure and bias, direct communication between clients and analyst should be avoided, except when necessary to answer questions regarding specific analytical procedures.

#### 3. SCOPE OF DOCUMENT

This QA Plan presents an overview of the essential elements of A & B Labs QA program. This plan is modeled along guidelines provided by the United States Environmental Protection Agency (USEPA), Texas Commission For Environmental Quality (TCEQ), American Industrial Hygiene Association (AIHA), National Environmental Laboratory Accreditation Program (NELAP), National Voluntary Laboratory Accreditation Program (NVLAP) and other federal and state regulatory agencies. These guidelines incorporate much of ISO/IEC 17025.

The QA program is designed to control and monitor the quality of data generated in A & B Labs. The program has four key elements.

- Demonstrating laboratory capabilities by providing information which documents the overall qualifications of the laboratory to perform the environmental analyses.
- Controlling laboratory operations by establishing procedures which measure laboratory and instrument performance on a daily basis.
- Measuring matrix effects to determine the effect of a specific matrix on method performance.
- Reporting appropriate QC information to enable the end user to assess the quality of data.

The specific procedures involved in implementing each aspect of the QA program are described in this document. The QA/QC policies and procedures described herein are designed to eliminate systematic errors and minimize the occurrence of other errors. These QA/QC policies and procedures must be coupled with the professional judgment of the technical staff in interpreting the events surrounding the generation of the final result to ensure that quality data is consistently produced, and decisions and corrective actions are fully documented.

This Quality Assurance Plan will be updated and revised when ever necessary to meet new regulatory guidelines. The Plan shall be reviewed at least annually and approved by management. Any changes to the plan must be approved by the President, Laboratory Director, and QA Officer. In instances where Quality Assurance Project Plans (QAPJP)s are required, A & B Labs will incorporate the elements of the QA plan while taking under consideration any specific requirements of the project.

All QAPJPs have the following components:

- Background and overall project objectives.
- List of required parameters.
- Quantity and frequency of samples to be taken.
- Sampling responsibility.
- Types of samples to be taken (i.e., surface water, groundwater, wastewater, soil, sludge, sediment, biological, air, dust, paint, etc.).
- Scheduling of project, including initiation and completion dates.

#### 4. **DEFINITION OF TERMS**

Quality Assurance: (QA): the total integrated program for assuring the reliability of data generated in the laboratory.

<u>Quality Control</u>: (QC): the routine application of specific, well-documented procedures to ensure the generation of data of known and accepted quality, thus fulfilling the objectives of the QA program.

Quality Assurance Plan: (QAP): an assemblage of policies, objectives, principles, and general procedures outlining the techniques by which the laboratory produces data of known and accepted quality.

<u>Standard Operating Procedure:</u> (SOP): a detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.

Quality Assurance Project Plan: (QAPJP): an assemblage of detailed procedures describing how the laboratory will generate data that meet the data quality objectives of a specific project.

<u>Legally Defensible Data</u>: data which are supported by a QAP and documentation adequate to reconstruct the analytical process. Legal defensibility is not dependent on the level of deliverables.

<u>Holding Time</u>: the period of time during which a sample can be stored after collection and preservation without significantly affecting the accuracy of the analysis.

## 5. PERSONNEL QUALIFICATIONS AND TRAINING

#### **Personnel Qualifications**

The laboratory shall employ sufficient and appropriate personnel to perform the tasks necessary to provide quality work. No employee hired can also hold a position with a client. If any associate or relative of an A & B employee submits work to the lab, that employee cannot knowingly work on those samples. Employees will be asked to read and sign an ethics statement.

All personnel must be qualified for the position that they hold in order to provide clients with satisfactory laboratory performance. Qualification requirements can be met through a combination of education, training and experience.

### **Technical Manager (President)**

The minimum qualifications for this position are a bachelor's degree, in chemistry or a related science, four (4) years industrial hygiene experience or certification by the American Board of Industrial Hygiene (ABIH), and five (5) years of environmental laboratory experience. Relevant academic experience may be substituted for work experience.

## **Quality Assurance Officer**

Minimum qualifications for this position are a bachelor's degree in chemistry or a related science and four (4) years of non-academic chemistry experience. In addition, documented training in statistics and quality control procedures are required.

## **Laboratory Director**

Qualifications for this position are a bachelor's degree in chemistry or a related science and four (4) years experience. A minimum of two (2) years experience in industrial hygiene and two (2) years experience in environmental analyses are required.

## Environmental Services Director/Polarized Light Microscopy Laboratory Manager

Qualifications for this position are a bachelor's degree in geology or related field and a minimum of 4 years experience. A TDH Asbestos Consultant License is required.

#### **PLM Laboratory Supervisor**

Qualifications for this position are a bachelor's degree in a science or related field and three years of environmental laboratory experience.

## Environmental (i.e., Organic, Inorganic, Microbiology) Department Manager

Qualifications for this position are a bachelor's degree in a science or related field and three years of relevant environmental laboratory experience.

#### **PLM Laboratory Analyst**

Qualifications for this position are a bachelor's degree in a science or related field, or experience and documented proof of ability to produce reliable analytical results. A part-time or non-degreed person may fill this position, however they must be well trained and work under the direction of the laboratory supervisor.

### **Environmental Laboratory Analyst**

Qualifications for this position are a bachelor's degree in a science or related field, or experience and documented proof of ability to produce reliable analytical results. A part-time or non-degreed person may fill this position, however they must be well trained and work under the direction of the laboratory supervisor.

## Mold Lab Technical Manager

Qualifications for this position are a bachelor's degree in a microbiology, biology, or related life science, with a minimum of 20 semester hours in microbiology and a minimum of two years of relevant experience. This individual must also be on-site 50 percent of the laboratory operating hours.

## Industrial Hygiene Analyst

Qualifications for this position are a bachelor's degree in a science or related field, or experience and documented proof of ability to produce reliable analytical results. The fiber counting microscopist is required to have completed a NIOSH 582 or an equivalent course.

## Project Manager

Qualifications for this position are a bachelor of science degree in a science or technical field and a general knowledge of the techniques and limitations of environmental testing. Good people skills are essential.

### **Training**

Training is a key aspect of quality assurance of the laboratory. It is the responsibility of the managers and laboratory supervisors to ensure that all laboratory personnel are trained properly in laboratory techniques, methods of analysis, and quality assurance procedures. Therefore, the manager, supervisors or their experienced designees, train all analysts to perform the analyses that are required of them.

Prior to performing any analyses all personnel receive a copy of the documents that they are required to read and understand. Such documents include: the Health and Safety Manual, the Quality Assurance Manual, copies of the methods, and copies of the current Standard Operating Procedures (SOPs), and the Ethics SOP.

The A & B QA program requires that all analysts have an analyst notebook. This notebook must contain a copy of all SOPs, training checklists and records for all tests the analyst is certified to perform. The training checklist has an area where all methods and SOPs that have been read and understood are to be listed. The training checklist requires that analysts date and initial each document that they have read and understand. By initialing each document the analyst is affirming that they have been trained in instrumentation, calculations, reporting, quality control, and safety set forth in the document.

The checklist is not complete until all methods that the analyst has been trained to perform are documented with demonstrations of proficiencies. The Lab Director and/or Quality Assurance Officer must initial these proficiency documents as acceptable before the training is considered complete. Management shall review the analyst training annually. The laboratory's QC checks and PT results serve as a continuing check on the analyst's performance and help identify ongoing training needs. Training is required prior to performing any new techniques or methods. Whenever deemed necessary, the QA Officer, manager, or supervisor may require re-training of any laboratory personnel. Training can be supplemented by attendance at seminars and courses.

## **Ethics and Data Integrity**

The laboratory managers and the supervisors must also ensure that all laboratory personnel receive training on ethics and data integrity as part of their new hire orientation. Receipt of ethics training shall be "signed off" by both employee and management representative. Data integrity training shall be reviewed yearly in one of the regular QA/Safety meetings. Correctness and completeness of data is checked in the

data reporting process. Personnel involved in fraudulent activities are subject to immediate dismissal. It is the responsibility of A & B employees to follow the rules set forth in the various manuals and SOP's that are specific to their tasks.

#### 6. RESPONSIBILITIES AND AUTHORITIES

Executing an effective QA program demands the commitment and attention of both management and staff. The QA Officer administers the QA effort at A & B Labs. The QA Officer reports directly to the President, and has the responsibility for overseeing and regulating all laboratory functions. The QA Officer operates independently of all areas generating analytical data to ensure complete objectivity in the evaluation of laboratory operations.

All analysts within the organization play a vital role in assuring the quality of their work. We believe that the success of A & B is dependent upon the continued commitment of all within the organization to a strong and viable QA Program. The responsibilities and authorities within the organization are described below. Job descriptions are also stored in the BTLIMS program.

### **Technical Manager**

At present, the President is also the Technical Manager. The Technical Manager provides overall direction and technical management of the laboratory. In the absence of the Technical Manager, the deputy Technical Manager is the Laboratory Director.

## Responsibilities

- Ensuring that the QA Officer has management support in the overseeing and regulating of laboratory functions.
- Assuring analysts are free from conflicts of interest and undue pressures.
- Conducting management reviews of the overall capabilities and qualifications of laboratory before commencing new analytical procedures or projects.
- Assessing sample preparation facilities, analytical equipment, personnel, and capacity to handle the quantities of samples, and any other requirements affecting the lab's ability to perform analyses, before commencing new projects.
- Ensuring that new SOPs are generated and approved before accepting samples for new projects.
- Conducting annual management reviews of the quality system to ensure continued suitability. This management review shall include:

- Suitability of policies and procedures
- o Management reports
- o Summarizing internal audit results
- o Corrective and preventive actions
- o Assessments by accrediting bodies or regulatory agencies
- o Results of proficiency tests and interlaboratory comparisons
- o Changes in the volume or type of testing
- o Client complaints and communications
- Staffing resources and training requirements
- Signing and authorizing release of reports

### **Authority**

- The Technical Manager has the authority to require that procedures be amended, discontinued suspended or repeated.
- The Technical Manager has the authority to suspend or terminate employees on the grounds of falsification of data or any other records, incompetence, or repeated non-compliance with QA procedures.

## **Laboratory Director**

The Laboratory Director conducts the daily operations of the laboratory.

## Responsibilities

- Help to set up and over see company budget and expenses.
- Help to determine instrument suitability and readiness.
- Ensure that sample turnaround and QA/QC meet client requirements.
- Track lab appearance and productivity weekly.
- Determine ways and means of improving company quality and efficiency.
- Respond to client inquiries or complaints.

## Authority

- The Lab Director has the authority to require that procedures be amended, discontinued suspended or repeated.
- The Lab Director has the authority to recommend suspension or

termination of employees on the grounds of falsification of data or any other records, incompetence, or repeated non-compliance with QA procedures.

- Authority comes directly from the President.
- Signing and authorizing release of reports

### **Quality Assurance Officer**

The QA Officer directs the QA effort within A & B. Under the management of the President, the QA Officer carries out the responsibilities of the office. In the absence of the QA Officer, the deputy QA Officer is the Laboratory Director.

## Responsibilities

- Developing and implementing a QA Program that ensures that all data generated at A & B Labs are scientifically sound, legally defensible, and of known precision and accuracy.
- Arranging for or conducting internal audits and inspections of the laboratory to identify potential problems and ensure compliance with written SOPs, reporting the results of those audits to the President, and applying corrective actions as needed to ensure compliance with the A & B QA Plan.
- Coordinating the distribution of Performance Evaluation (PE) samples, evaluating the results of those samples, and reporting the results to the President, then applying corrective actions as needed to ensure that the laboratory is able to generate data that meet the data quality objectives defined in the A & B QA Plan.
- Handling interactions between the laboratory and clients with QA concerns.
- Coordinating certification programs.
- Assisting analysts in the writing of SOPs.
- Maintaining records and archives of all QC data, PE results, audit comments, and customer inquiries concerning data quality.
- Assuring that the analysts have access to current SOPs.
- Preparing QA Project Plans when needed.
- Updating the A & B QA plan whenever necessary, and revising the plan annually.
- Assuring analysts accessibility to the QA plan.
- Signing analytical reports and authorizing their release.

### **Authority**

- The QA Officer has the authority to require that procedures be amended, discontinued suspended or repeated.
- The QA Officer can also make recommendations regarding termination of employees for falsification of data or any other records, incompetence or non-compliance with QA procedures. Non-compliance with QA procedures includes: failure to implement corrective actions, a consistent pattern of failure to implement effective corrective actions, and discontinuation of implemented corrective actions.
- Authority comes directly from the President.

### **Laboratory Managers and Supervisors**

The manager and supervisors who direct the analytical work at the laboratory are directly responsible for ensuring that all employees reporting to them are well trained and complying with the A & B QA Plan.

## Responsibilities

- Assuring that all employees reporting to them have completed the training program, and monitoring training on an on-going basis.
- Actively supporting the implementation of the A & B QA Plan.
- Maintaining accurate SOPs and enforcing their use in the laboratory.
- Maintaining a work environment that emphasizes the importance of data quality.
- Handling laboratory and client communications that require management input.

## **Authority**

The manager and supervisors of the laboratory have the authority to accept or reject data based on compliance with well-defined QC criteria. In addition, the manager and supervisors, with the approval of the QA Officer, can accept or reject data that fall outside of established QC guidelines if, in their judgment, there are technical reasons which warrant the acceptance or rejection of the data. They may also require re-training at anytime that they deem necessary. Their authority comes directly from the President.

#### **Laboratory Personnel**

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the A & B QA Plan.

### Responsibilities

- Having a working knowledge of the A & B QA Plan.
- Ensuring that all work is generated in compliance with the A & B QA Plan.
- Performing all work according to written SOPs.
- Providing the QA Officer, Laboratory Manager, Supervisor and/or Technical Manager with immediate notification of quality problems.
- Additionally, all analysts are responsible for assuring that the housekeeping in their work areas is adequate to prevent contamination of samples and to minimize fire and safety hazards.

## **Authority**

 Laboratory personnel may on their own authority accept or reject data based on defined QC criteria. Acceptance or rejection of data that fall outside of established QC Guidelines must be approved by the QA Officer and laboratory management.

## **Project Manager**

The Project Manager interfaces with the client to ensure that client goals are being met.

## Responsibilities

- Answering technical questions.
- Ensuring that customer turnaround times are met.
- Ensuring that client analytical requests are accurately logged in.
- Giving out pricing information and quotes.
- Resolving ambiguities between A & B and Client.
- Checking reports for accuracy and completeness
- Signing and authorizing release of reports

### **Authority**

• The Project Manager reports to the Lab Director. He/She has the authority to approve and sign analytical reports. He/She has the authority to give pricing and make quotes.

### **Customer Service Representative**

• This person must act as a liaison between the laboratory and the clients. He/She must coordinate the efforts of sample collection, sample receiving, and sample custody. In doing so, this person assures a positive interaction between clients and the laboratory.

## Organizational and Management Structure

The entire laboratory falls under the leadership of the Technical Manager. All laboratory managers report directly to the President. The QA Officer, as stated previously, is independent of the analytical areas and also reports directly to the President. Additional information concerning the organizational structure of the laboratory can be found on the organizational chart, which is in Appendix I.

## Interdepartmental Communication

Regular meetings facilitate interdepartmental communication. A supervisors meeting is held every two weeks to inform management of news, problems, new projects, and completion of old projects. QA reports to management are given at this time. Monthly Safety/QA meetings are held with the entire staff to inform employees of any changes decided on in management meetings, news, problems, corrective actions that involve general procedures, and results of PT studies. Employees are encouraged to take the floor and air any problems or suggestions.

### 7. EQUIPMENT

Laboratory equipment and instrumentation necessary to performing required analyses is made available to authorized laboratory personnel. Instructions for use of instruments/equipment in the form of instrument manuals, manufacturers instructions and SOPs are available in a central location in each lab. Whenever new equipment is purchased, it is not put into use until it has been inspected and calibrated. Whenever previously owned equipment is purchased, it is not put into use until it has been inspected by a trained technician and calibrated.

Laboratory instrumentation is logged into the BTLIMS Labware module. The instruments are assigned IDs. Model number, serial number, manufacturer, date manufactured, date purchased, date put into service, location, and primary operator are included in the lab ware records.

Instruments that are not functioning within acceptance criteria are pulled out of service and clearly labeled (with a red dot) to indicate status. When moving equipment or instruments for repair or maintenance, care must be taken to prevent unnecessary contamination and agitation. Review instrument manuals for manufacturers instructions, keep instrument level, restrain moving parts, and, if necessary plug lines.

Following is a summary of the instruments used at A&B Labs.

## **VOC Laboratory:**

HP 5890 GC with HP 5970 MS and Tekmar LSC 2000 ALS 2016 HP 5890 GC with HP 5971 MS and Arcon 3000 HP 5890 GC with HP 5971 MS and Entek preconcentrator HP 5890 GC/5970 MS

## **GC Laboratory:**

HP 5890A GC with FID/FID (1) HP 5890 GC/HP 5891 MS (2) Varian 3400 ECD/ECD (2) Varian 3600 PID/FID Varian 3400 PID/FID HP 5890 ECD/ECD (2) HP 5890 NPD/NPD HP 5890 GC/FID (2)
Orion AF7LC Coulometric Titrator
Photovolt Aquatest IV Coulometric Titrator

#### **Extraction Laboratory:**

Glas-Col 3D Shaker
Dionex ASE3000 Accelerated Solvent Extraction System
Sonicator (2)
Turbovap
Eberbach Shaker

#### **Metals Laboratory:**

PE 5100ZL Furnace AA PE Optima 3300DV ICP PE 5000 AA Cold Vapor

### **Metals Digestion Laboratory:**

Precision Water Bath for Mercury digestions Environmental Express Hot Block Digester (2)

## Wet Laboratory:

PE Scanning IR

Foss Tecator Kjeldahl Digester

Glaston Mididist for Phenol, Ammonia, Fluoride Distillations

Glaston Mididist for Cyanide

Hach Odyssey Spectrophotometer

Turner Fluorometer 111

Koehler Penske-Martin Flash Point Apparatus

Radiometer pH meter (2)

YSI DO meter

Metrohm 605 pH meter (2)

Incubators and Ovens

## **HPLC Laboratory:**

Varian 9090 Sampler with Varian 9010 Solvent Controller and Waters Model 481UV and Model 420 Fluorescence Detectors

Varian 9095 Sampler with Waters Solvent Controller and Waters Model 481 UV and Model 420 Fluorescence Detectors

Dohrman TOC

Thermo Glas TOX Analyzer Dionex IC20 IC Dionex 4000i IC

## Other laboratory instruments include:

Orion Model 420A pH Meter
Orion Model 80 Ion analyzer
Varian DMS 90 UV/VIS Spectrometer
Mettler AE 163 Balance
Various top loader balances
Various water baths
Various centrifuges
Olympus BH-2 Polarized Light Microscope
Nikon Labophot Polarized Light Microscope
ERC Muffle Furnace

#### 8. SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, and therefore the integrity of the sample collection process is of concern to the laboratory. Samples must be collected in such a way that no foreign material is introduced into the sample and no material of interest escapes from the sample prior to analysis. To ensure sample integrity, the following must be considered:

- Samples must be collected in appropriate containers. In general, glass containers are used for organic parameters and polyethylene containers are used for inorganic/metal environmental parameters.
- Samples for industrial hygiene parameters are collected in sorbent tubes, passive monitors, filters, wipes and bottles.
- An ample supply of new sample containers and preservatives must be available, so the appropriate containers and preservatives are always utilized.
- The sample container must be properly cleaned to ensure that the sample is not contaminated during the collection.
- Unless certified clean, all new lots of sampling media must be tested by the laboratory for unacceptable contamination prior to use in the field.
- Samples must be preserved appropriately to minimize the loss of materials of interest due to adsorption, chemical or biological degradation, or volatilization.
- To avoid damage and deterioration, sample containers must be transported in rigid packages, glass bottles protected from bumping, with ice or blue ice when necessary
- Appropriate volumes of sample must be collected to ensure that the required detection limits can be met and quality control samples can be analyzed.
- Samples must be properly shipped to the laboratory, in the appropriate

time frame, to ensure that holding times for the analytes can be met.

### **Sampling Program Operations**

Environmental sampling is not part of A&B Laboratory's routine operation. Client must provide specific sampling requirements if A&B is required to oversee sampling activities. Specific sampling program operations will be defined in the QAPJP when a project is contracted by A & B Labs that includes sampling. Whenever deviations from the sampling plan are required by client or field conditions, notations shall be made on the chain of custody or on the Job folder or as an addendum to the chain of custody. Relevant environmental conditions are recorded on the chain of custody if required by the project plan.

When collecting samples from effluent streams, sampler will provide the following information on the chain of custody:

Sampler's name and company
Sample location
Date and time of sample collection
Number of bottles collected and preservative used
Type of sample (composite/grab)
Tests required
Temperature, if required by client
D. O., if required by client
pH, if required by client
Flow, if required by client

## Sample Containers

A & B can provide proper containers and preservatives for most analyses. Clean, new containers are supplied. When requested, A & B can provide certified precleaned sample containers for trace analyses. The type of container necessary is determined by the test and reporting limit required. A & B does not make a practice of providing sampling media for IH sampling but recommendations are made so that proper sampling media can be selected from vendor catalogs.

## **Holding Times**

The EPA has established holding time requirements for some analyses. These

holding time requirements are listed in the Appendix II (attached), along with container and preservative requirements. A & B Labs follows the holding times given in Update 1, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, November, 1990.

### **Sub-Samples**

When samples are divided for analytical purposes, care must be taken to ensure that the portion taken and the portion left behind are representative of the sample as a whole. When necessary sample shall be subjected to particle size reduction to ensure representative sub-samples. The portions removed from the original container along with any digestions or extracts made from them must be clearly labeled to avoid any confusion regarding identity. All sample portions must be transported to and from coolers in a safe manner using appropriate carriers to avoid breakage. Applicable SOPs must be followed.

## 9. SAMPLE CUSTODY

### Receipt

Sample receiving, log in, and storage, flow is as described in Figure 9-2. Samples should arrive at A & B with a sample custody form generated in the field. The chain of custody form should contain information about sampling date and time as well as sample identifiers and tests required. When not available, a letter detailing the same information can be substituted. The chain shall be signed by the sampler as well as by the laboratory representative. An example of the environmental COC record is given in Figure 9-1. The analytical tests requested are reviewed by receiving personnel to determine if the tests are adequately defined, documented and understood. This may involve checking with analysts or management.

### Acceptance

A sample condition checklist is completed for each set of samples that is received by the lab. This form gives such information as temperature, preservation, matrix, breakage, and gives the log in personnel an area to write any special instructions from the client that are not included on the chain. Signing the sample checklist indicates acceptance of the sample. A copy of this form is sent with the analytical report and is kept in the sample folder. See figure 9-3. Upon acceptance all samples are assigned and labeled with a unique sample identification number and are logged into the Laboratory Information Management System (LIMS). All relevant SOPs are followed.

## Sample Rejection

Samples may be rejected for the following reasons:

- No chain of custody.
- No unique sample numbers or other ID.
- Improper containers.
- Excessive amounts of sample material.
- Holding time has been exceeded.

- Insufficient amount of sample.
- Improper preservation.
- Sample leakage

If samples are rejected due to any of the conditions listed above, the client is notified immediately and given the opportunity to correct the problem, void the samples, or proceed with sample analysis. Once the problem has been corrected, the samples are accepted for login.

If samples show signs of damage or contamination, the client is also notified immediately and given the opportunity to correct the problem. Insufficient chemical preservation shall be noted on the sample condition checklist. Extra preservation may be added by analyst to hold the sample for analysis if the sample is deemed to be unaffected by transport.

All communications with the client about their samples are noted and kept with the sample folder. Samples analyzed despite quality concerns, will be flagged and the concerns will be addressed in a report footnote or a case narrative.

## Storage

In order to protect samples from deterioration, contamination, loss or damage during storage, once accepted by the laboratory, samples are to be kept in secure storage, properly preserved. Only the log in personnel and the analysts conducting the analyses may handle samples. After log-in the samples are stored according to the conditions specified by preservation protocols. Samples that require thermal preservation shall be stored under refrigeration that is ±2° of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable. Refrigerators maintained at 4°C are monitored each workday. Samples are stored away from all standards, reagents, food, and other potentially contaminating sources. Samples shall be stored in such a manner to avoid cross contamination. Samples with short hold times are delivered to appropriate analyst by receiving personnel. All volatile analysis sample portions and microbiology samples are stored in separate refrigerators from other samples to avoid contamination, and all bulk samples are stored separately from air samples. Bulk asbestos samples are placed in the designated asbestos sample storage cabinet. The sample storage location is

entered into the LIMS during sample login.

## Sample Disposal

All environmental samples are retained for a minimum of 30 days. All industrial hygiene samples are retained a minimum of 90 days. When possible sample are kept under refrigeration until they are ready for disposal. Samples that are past their holding times may be stored at room temperature.

All samples, digestions, and sample extracts are disposed of according to procedures that meet the Federal and State regulations. Excess and hazardous samples are at times returned to the client.

## FIGURE 9-1

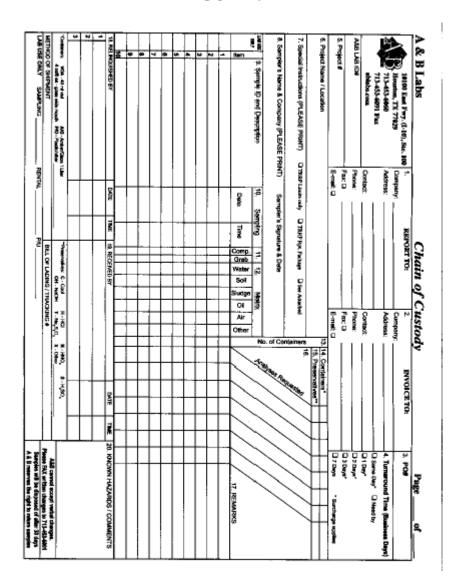
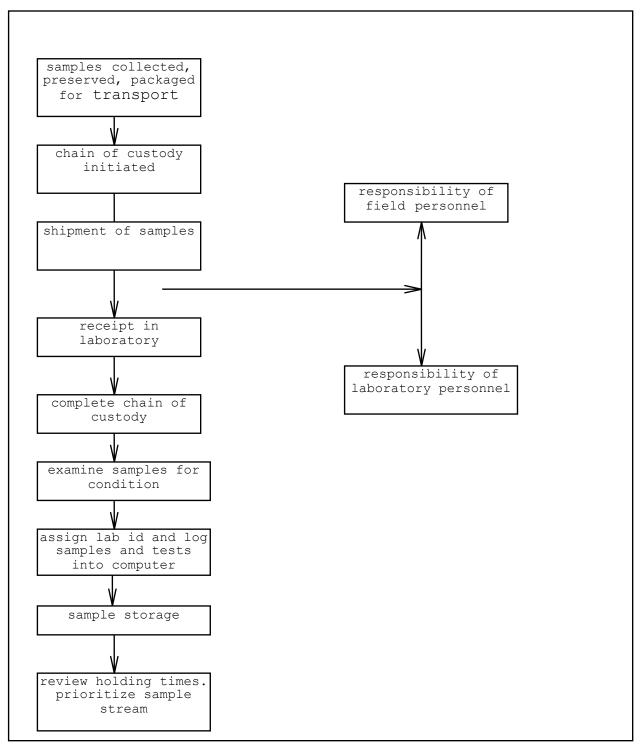


Figure 9-2 Sample Collection, Transport, and Holding Flow Chart



# Figure 9-3 Sample Condition Checklist

| Lab ID#:             |   | Date Received:                         | Time Received: |      |       |  |  |  |
|----------------------|---|--|----------------|------|-------|--|--|--|
| Company Name:        |   |  |                |      |       |  |  |  |
| Proj                 | ect:  |  |                |      |       |  |  |  |
| Ten                  | perature:   | Sample pH:                             |                |      |       |  |  |  |
| Check Points         |   |  |                |      |       |  |  |  |
|                      |   |  |                | Yes  | No    |  |  |  |
| 1.                   | Cooler Seal present and sign                        | ned.                                   |                |      |       |  |  |  |
| 2.                   | Sample(s) in a cooler.                              |  |                |      |       |  |  |  |
| 3.                   | If yes, ice in cooler.                              |  |                |      |       |  |  |  |
| 4.                   | Sample(s) received with chain-of-custody.           |  |                |      |       |  |  |  |
| 5.                   | C-O-C signed and dated.                             |  |                |      |       |  |  |  |
| 6.                   | Sample(s) received with sign                        | ned sample custody seal.               |                |      |       |  |  |  |
| 7.                   | Sample containers arrived intact. (If No comment)   |  |                |      |       |  |  |  |
| 8.                   | Matrix: Water Soil Lig                              | uid Sludge Solid Cassette Tube         | Bulk Badge     | Food | Other |  |  |  |
| 9.                   | Samples were received in ap                         | opropriate container(s)                |                |      |       |  |  |  |
| 10.                  | All samples were tagged or labeled.                 |  |                |      |       |  |  |  |
| 11.                  | Sample ID labels match C-O-C ID's.                  |  |                |      |       |  |  |  |
| 12.                  | Bottle count on C-O-C matches bottles found.        |  |                |      |       |  |  |  |
| 13.                  | Sample volume is sufficient for analyses requested. |  |                |      |       |  |  |  |
| 14.                  | Samples were received with                          | in the hold time.                      |                |      |       |  |  |  |
| 15.                  | VOA vials completely filled                         |  |                |      |       |  |  |  |
| 16.                  | 5. Sample accepted.                                 |  |                |      |       |  |  |  |
| Cor                  | nments: Include actions tak                         | ten to resolve discrepancies/problems: |                |      |       |  |  |  |
|                      |   |  |                |      |       |  |  |  |
|                      |   |  |                |      |       |  |  |  |
|                      |   |  |                |      |       |  |  |  |
|                      |   |  |                |      |       |  |  |  |
| Check in by/date:    |   |  |                |      |       |  |  |  |
| LIMS log in by/date: |   |  |                |      |       |  |  |  |

## 10. CALIBRATION PROCEDURES AND FREQUENCY

### **Instrument Calibration and Tuning**

To ensure accuracy of the test method, each instrument system used to generate analytical data must be calibrated. Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet reporting limits. Each instrument is calibrated with standard solutions, prepared from certified stocks, appropriate to the type of instrument, the established linear range, and the analytical method requirements. The frequency of calibration and calibration checks is determined by the manufacturer's guideline, the analytical method, EPA requirements, or the requirements of special contracts. The number of standards required for a calibration curve is often specified in the analytical method. When not specified, the minimum number of standards used is two in addition to a blank. For instruments that require single point calibration, a standard must be run with each analytical batch at the limit of quantitation.

If initial calibration does not meet acceptance criteria, corrective action must be performed and all associated samples re-analyzed. If re-analysis is not possible, data associated with an unacceptable initial instrument calibration shall be reported with appropriate data qualifiers.

#### **Calibration Records**

Each instrument has a calibration notebook and/or computer log to maintain calibration and standardization records. The following information is included:

- Type of instrument.
- Manufacturer.
- Serial number or inventory number.
- Frequency of calibration and standardization.
- Calibration and standardization.

Blanks, standards and samples analyzed.

Following are some general rules for instrument calibration.

## Gas Chromatography/Mass Spectrometry (GC/MS)

- Each day prior to analysis of samples, the instrument is tuned with bromofluorobenzene (BFB) for volatile compounds and decafluorotriphenylphosphine (DFTPP) for semi volatile compounds. The instrument must be tuned every 12 working hours. No samples are analyzed until the instrument has met the tuning criteria set forth in the specific method. If tuning criteria cannot be met, the instrument must be re-calibrated.
- A minimum of three internal standards and three surrogates are selected to spike each blank, standard and sample.
- Each parameter of interest is calibrated and an average response factor is calculated or, if linear regressions is used, a correlation coefficient is calculated..
- The relative standard deviation of the response factors for each compound must be  $\leq 20\%$  or the correlation coefficient must be  $\geq 0.990$ .
- An Initial Calibration Verification standard (ICV), from a second source standard, is performed after the initial calibration. Acceptance criteria is ±25% of expected value. Re-calibration must be performed if ICV fails acceptance criteria after corrective action and repeat of ICV.
- Continuing Calibration Verification standard is analyzed daily before sample analysis every 12 hours. If CCV does not pass acceptance criteria of ±20% and corrective action does not result in a passing CCV, instrument must be re-calibrated.
- Each response of the Calibration Check Compounds must fall within ± 20% of initial calibration response factor for each compound in the calibration check compound list. If CCC compounds do not pass acceptance criteria, instrument must be re-calibrated.

• A method blank is run after each calibration verification and with each preparatory batch. No analytes should be detected ≥1/2 RL For common laboratory contaminants, no analytes should be detected ≥RL.

### Gas Chromatograph (GC)

- GCs with detectors other than MS (FID, NPD, ECD, PID) are calibrated with a blank and five standards, surrogate standards, and internal standards where appropriate.
- An Initial Calibration Verification (ICV) from a second source standard is performed following initial calibration. All analytes must be within ±20% of expected value (%D). Re-calibration must be performed if ICV fails acceptance criteria after corrective action and repeat of ICV.
- A calibration verification standard is run daily before the start of each analysis, after every ten samples, and every 12 hours. Recalibration is done if the CCV is off more than ± 20%. Run ends with a CCV.

## Graphite Furnace Atomic Absorption Spectrometer (GFAA)

- A minimum of three (3) standards for each metal is analyzed daily within the EPA recommended linear range.
- The absorbance of each standard is recorded and regressed. Acceptance criteria for the regression is r≥0.995. A major deviation of absorbance from one day to the next, even if the standards are linear, indicates external problems that must be corrected. (i.e., lamp is losing energy or bad standard).
- Prior to analysis, a QC standard from a different source than the calibration standard (ICV) is analyzed. Recovery must meet EPA recommended limits.
- Perform CCV and CCB (continuing calibration blank) every 10 samples and end run with CCV and CCB.

# Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP)

- The instrument is calibrated before each use with a minimum of three (3) standards and a blank.
- An external reference standard (ICV) is run prior to the samples and the recovery must meet EPA recommended limits.
- CCV and CCB shall be run every ten samples.
- Recalibration must be done if the ICV is off more that  $\pm 5\%$  or the CCV is off more than  $\pm 10\%$ .
- Run ends with a CCV and CCB.

#### Other Laboratory Instrumentation

### Xertex Dohrmann Carbon Analyzer

- A one (1) point calibration is run daily.
- A check standard of a different concentration and source and a blank are run before samples are analyzed.

# Waters High Performance Liquid Chromatograph

- The instrument is calibrated with a minimum of three (3) standards and a blank. An initial calibration verification standard and a blank are run before samples are analyzed. Re-calibration is performed if the ICV is more than ±20% different from expected value.
- CCV is analyzed every ten samples and at the end of the run. CCV %drift/difference for any individual analyte must be <20%. If CCV fails to meet acceptance criteria, take corrective action and repeat. Repeat sample not bracketed by passing CCVs. Re-calibrate if unable to get passing CCV.</li>

- The instrument is calibrated with a minimum of three (3) standards and a blank. An initial calibration verification standard and a blank are run before samples are analyzed. Re-calibration is performed if the ICV is more than ±20% different from expected value.
- CCV is run every ten samples and at the end of the analytical sequence. CCV %drift/difference for any individual analyte must be <20%D. If CCV fails to meet acceptance criteria, take corrective action and repeat. Repeat sample not bracketed by passing CCVs. Re-calibrate if unable to get passing CCV.

### Koehler Penske-Martin Flashpoint apparatus

• A xylene standard is run in duplicate to calibrate this instrument. The thermometer is checked annually against a certified NIST-traceable thermometer.

### **Hach Spectrometer**

• A minimum of three (3) standards and blank are used to generate data for a linear regression.

# Fisher Accumet pH Meter Model 600

• Two (2) standard buffers encompassing the expected range of the samples are run. Additional buffers are run to bracket the samples.

# Orion Model 80 Ionalyzer

• A minimum of three (3) standards and blank are used to generate data for a linear regression.

# YSI Model 54A Oxygen Meter

• Meter is air calibrated according to temperature and the scale on the back of the instrument.

Mettler AE 163 Analytical Balance

Sartorius 1200 Top Loading Balance Denver P-2000 Top Loading Balance Denver APX-602 Top Loading Balance Ohaus Top Loading Balance Acculab V-3 Top Loading Balance

- Daily calibration checks are made according to SOP using a minimum of two NIST traceable weights.
- Annual service is performed by an outside contractor with ISO certification using NIST traceable weights.

All thermometers are checked annually against a certified NIST-traceable thermometer. Results must be within the manufacturer's specifications. If not within specifications, the thermometers are flagged with the deviations, and replaced. No flagged thermometers are used unless it is an emergency. If a flagged thermometer is used, the temperature is adjusted accordingly.

Pipetters are calibrated quarterly. Delivery must meet manufacturers specifications or a correction factor must be applied.

Hood face velocity must be measured annually.

Where calibrations give rise to correction factors, the equipment or instrument shall be marked with the correction factor or each copy of software used for calculation is updated as appropriate.

#### 11. ANALYTICAL PROCEDURES

### **Adoption**

Most analyses performed by A & B Labs are driven by regulatory concerns. Therefore, methods adopted by A & B Labs predominantly originate from regulatory agencies, and are strictly adhered to until a revision is received from the regulatory agency. Generally the methods are those specified by the U. S. EPA and other federal and state agencies, as provided in the following references:

- 40 CFR 136
- Test Methods for Evaluating Solid Waste, SW 846
- NIOSH Manual of Analytical Methods
- ASTM
- Standard Methods for the Examination of Water and Wastewater, APHA,
- Methods for Chemical Analysis of Water and Waste, EPA-600/4-79-020
- Methods for Determination of Organic Compounds in Drinking Water, EPA
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA
- Biological Analytical Manual, FDA
- AOAC
- <u>Diagnostic Microbiology</u>, Bailey and Scott
- Compendium of Methods for the Microbiological Examination of Foods, APHA

When more than one analytical procedure is available, the Lab Director/designee decides which is adopted based on criteria such as availability of standards, reagents, and instrumentation. Training for new methods is assigned and monitored by the Department Manager or the Lab Director.

# **Standard Operating Procedure**

Analytical procedures for the analysis of samples are documented with a SOP (Standard Operating Procedure) for the particular parameter needed. These SOP's must contain enough information to enable a 'walk-up performance' of the method. Content of SOPs should be consistent with NELAC standards. Included in the SOP must be at a minimum:

Application of the method.

Summary.

Safety.

Sample preservation and holding times.

Apparatus.

Reagents and standards and preparation.

Sample Preparation or extraction method.

Procedure.

QA/QC requirements

Calculations.

Reporting.

Waste Management

Pollution Prevention

References.

A SOP format is given in appendix III. When at all possible, this SOP format is to be followed. SOPs are also generated to define procedures for non-analytical activities such as assessing data quality, data validation and reporting, and manual integrations. If the subject of the SOP does not lend itself to the given format, mark non-relevant sections N/A.

Occasionally, it may be necessary to deviate from the documented policies and procedures to provide a client with required information. If a deviation is necessary, the client must be informed in writing that his requirements will cause a deviation from the approved method. Additionally, they must be informed as to what the deviation is, and why it is necessary. The Technical Manager or QA Officer will then approve the changes in the method, after consulting with the analyst responsible for performing the procedure. The method on the final report will be identified as Modified.

#### **Demonstration of Performance**

Although the methods in use are generally mandated by regulatory requirements, performance proficiency of these validated methods to cover the concentration and matrix of interest must be demonstrated. New tests or methods must be evaluated to determine limit of detection, limit of quantitation, or range of applicability, such as linearity.

Methods must not be used until competence has been demonstrated by the analyst. An Initial Demonstration of Proficiency may include method-mandated analyses or may include analysis of four midrange Laboratory Control Samples (LCS). Statistic generated by the four LCSs must meet method or lab requirements. Method mandated Demonstrations of Proficiency should be described in individual SOPs. Analyst must also perform a linear calibration and method detection limit (MDL) studies. Whenever there is a change in methodology, instrumentation or personnel, linear calibration and MDL studies must be reestablished. MDL studies must also be established for each matrix analyzed under a particular methodology. For methods with stated MDLs, demonstration of ability to achieve such MDLs is required. Continuing demonstration of proficiency is provided by routine QC checks and blind performance testing samples (PT samples).

### Reviewing and Revising

Each supervisor shall review SOPs at least annually. The supervisor shall determine if the method is still meeting required detection limits, linearity, and regulatory concerns. If a new method or modifications to the method need to be made, the quality assurance officer and laboratory director shall be notified. The details of any changes shall be discussed in the next management meeting and a new SOP shall be written. Any changes to the method mandate a new MDL study and LCS validation before the method can be put into service. New SOPs must be approved and signed by the Lab Director and the QA Officer before being issued for use.

A summary of the tests performed by A & B Labs is listed in Appendix VI.

### 12. INTERNAL QC CHECKS

### Assuring the Quality of Test Results

A&B Lab implements its QC procedures to monitor the validity of the tests being done. This monitoring is planned and reviewed and may include, but not be limited to, the following:

- Regular use is made of certified reference materials and/or internal quality control using secondary reference materials. These materials may be used as calibration standards, calibration verification, or positive control.
- Participation in interlaboratory comparisons or proficiency-testing programs is part of routine analysis.
- Replicate tests using the same or different methods can be used to verify analyte identity or instrument performance. Example: Ammonia electrode may be checked by simultaneous analysis using indophenol method.
- Retesting of retained samples tests not only instrument stability but may also be used instead of standards or duplicates for tests that have no certified standards. Example: mold or asbestos
- Correlation of results for different characteristics of a sample (for example total phosphate should be greater than or equal to orthophosphate is a check for reasonableness of results.
- The QC program monitors data quality with internal QC checks. The QC checks help determine if operations are in control and if the sample matrix is affecting the data being generated.

#### **Blanks**

In order to effectively monitor QC, the analysis of blank samples is essential. A method blank is analyzed to assess the level of background or contamination that exists in the analytical system and which might lead to reporting elevated concentration levels or false positive data. A method blank consists of reagents, specific to the method, which are carried through every aspect of the procedure. The method blank is analyzed after instrument calibration. A method blank is analyzed specific to each matrix and with each batch of samples analyzed. An example of a method blank is the digestion blank initiated with each batch of samples digested for metals analysis. The acids used in the digestion procedure

are added to DI water or clean sample matrix in the same concentration as would be used in a sample. The blank is digested with the samples and under the same conditions. The digestion blank is then analyzed in the same run as the sample batch of which it is a part.

Various field blanks may also be run to assess the possibility of contamination in the sampling, transport, and operations. These blanks (equipment blank, trip blank, or field blank) are initiated by the client and treated like a sample.

It is not A & B policy to correct sample analytical results for contamination found in a blank. Reportable concentrations of target compounds in a blank necessitates the reanalysis of the blank and may require a repeat preparation of the entire batch. If this is not possible the data is qualified and the blank concentrations are reported. If at the clients request or if the method requires it, blank correction is performed, the report is clearly labeled as such.

### **Duplicates**

A duplicate is a sample split in the lab, prepared, and analyzed separately. Duplicates are analyzed to monitor precision or reproducibility on an on-going basis. In general, duplicates are analyzed on a 10% basis for inorganic and metal parameters and on a 5% basis for organics. However, frequencies for duplicate analyses can be adjusted to meet requirements for other regulatory agencies. Matrix spike duplicates or laboratory control samples duplicates are often done instead of sample duplicates. The organics department almost exclusively uses MSD or LCSD to monitor precision. The precision is expressed as a relative percent difference (RPD). This is the difference between the two numbers divided by their average (times 100 to convert to percentage). Statistical analysis is performed twice per year on the RPDs to determine acceptable control limits. Analyses outside the control limits are repeated.

Field duplicates are two samples taken at the same time and under the same conditions and treated as two separate samples. Field duplicates are useful for assessing the precision of the sampling technique. At least one field duplicate is advised per sample batch. Field duplicates must be initiated by the client.

# Replicates

Replicates are multiple measurements on a prepared sample. Replicates are used

to monitor precision and are useful to isolate instrument and operator precision. Re-runs of digested or extracted samples and re-counts of microbiological plates and asbestos PCM slides are replicates. Intra-laboratory duplicates are replicates read by different analysts. Inter-laboratory duplicates are samples shared with another lab and analyzed by different analysts for QC purposes.

### **Laboratory Control Samples**

Laboratory control samples (LCS) are laboratory generated samples used to monitor the laboratory's day-to-day accuracy in the performance of routine analytical methods. Each LCS consists of control matrix (de-ionized water, Ottawa sand, IH media, or other appropriate media) that is spiked with a representative group of the analytes of interest. If analytes of interest cannot be spiked into a clean control matrix, a cleanup procedure is performed on the matrix. The matrix is then analyzed to determine method suitability. If suitable, the matrix is spiked with the analytes of interest. If unsuitable, further cleanup is performed, or a sample matrix which has been determined to be free of the analytes is spiked with the analytes of interest. Laboratory control samples may be purchase commercially or a PT sample of adequate range may be used.

LCS's are prepared with each batch of samples and analyzed prior to the analysis of samples. Control criteria must be satisfied before samples are analyzed. LCSs are sometimes randomly placed within a batch of samples to monitor continued control.

Accuracy is expressed as a percent recovery. That is measured value divided by true value times 100.

$$\% \operatorname{Re} c = \frac{LCSr}{LCSt} * 100$$

where LCSr is recovered or measured concentration LCSt is true or assigned concentration.

Statistical analysis is done on the percent recoveries to determine acceptable limits. An out of control LCS necessitates reanalysis of the sample set.

# Matrix Spikes and Matrix Spike Duplicates

A Matrix spike (MS) is a sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure and the

recovery of the analytes is calculated. One MS per batch is required for all parameters, or on a 10% basis for metals and 5% basis for organics.

A Matrix Spike Duplicate (MSD) is a sample that is divided into two separate aliquots, each of which is spiked with known concentrations of analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as Relative Percent Difference (RPD) and percent recovery.

Control limits are established with statistics. An out of control recovery of a MS or MSD requires a bench spike (post digestion spike). This a spike added at the instrument to prepared sample. Care should be taken to avoid diluting the sample. If the recovery of the bench spike is also out of control then there is matrix interference and the numbers are reported with a qualifier. If the use of a bench spike is not possible but examination of chromatograms or other data gives information about sample matrix that infers matrix interference, then the data is reported with a qualifier. An example of this would be chlorine in a sample for sulfide.

# **Surrogate Recoveries**

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis. Results are reported in terms of percent recovery. Acceptance limits are established by a statistical analysis. An out of control result necessitate a reanalysis of the sample. At times the surrogate is lost due to non target peaks co-eluting with the surrogate. In these cases the sample is reported with a qualifier. If it is not possible to repeat the analysis, the results are reported as an estimate for the compounds affected by the out of control surrogate.

# **Proficiency Testing**

A & B Laboratories participates in a number of proficiency testing programs. An outside contractor administers proficiency testing. Unknowns received from the contractor are prepared and analyzed as samples. No more QC is allowed in the analysis than would be done in a typical analytical run. The results are sent back to the testing firm, and an evaluation of the lab's performance is sent back to the lab. The PT samples give an external gauge of analyst and laboratory

Section No. 12 Revision No. 22 Date 2/2006 Page 43 of 74

performance and verification of analytical method.

### 13. ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY

### **Data Quality Assessment**

The effectiveness of a QA program is measured by the quality of data generated by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, comparability, and completeness. These terms are described as follows:

<u>Precision</u> is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of reference material or environmental samples. Precision is expressed as Relative Percent Difference.

RPD = 
$$\frac{200 \mid (X_1 - X_2) \mid}{X_1 + X_2}$$
 Where  $X_1$  and  $X_2$  represent duplicate results.

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using LCS, standard reference materials, or spiked environmental samples. Accuracy is expressed as Percent Recovery.

%Rec. = 
$$\frac{100(R_x)}{R_{tv}}$$
 Where  $R_x$  represents any spiked result and  $R_{tv}$  represents its true value.

Acceptance Limits for precision and accuracy are set at two standard deviations  $(\pm 2\sigma)$  from the mean of the results for warning limits, and three standard deviations  $(\pm 3\sigma)$  from the mean of results for control limits. Acceptance limits are calculated from the accumulated quality control data. The LIMS can calculate limits for the parameters in each test. This calculation can also be done easily in excel after copying the QC data from the LIMS to a spreadsheet. These calculations are made at a minimum of twice per year. Control charts are also made at this time. New control limits are entered into the QC database and print out on the reports. As new LCS and LCSD data are entered into the QC spreadsheet it is compared to the control limits calculated. Having the quality data in a graphical form makes it easier to spot trends. Acceptance limits are used as a tool to determine if the analytical system is in control. If any of the following situations occur, the system may be unstable or out of control:

One or more points fall outside the control limits (mean  $\pm 3\sigma$ ).

Two points, out of three successive points, are between  $\pm 2$ to3 $\sigma$  or beyond. Four out of five successive points are between mean+1 $\sigma$  and mean+2 $\sigma$  or mean-1 $\sigma$  and mean-2 $\sigma$ .

Eight successive points are on the same side of the central line (mean).

Whenever one of these conditions is detected, the laboratory must investigate to determine the cause and document actions taken. If recovery falls outside of the control limit the associated samples would typically be re-analyzed. If there is insufficient sample or holding time, data qualifiers are applied to the QC batch.

Trending Trends should be investigated because they may indicate a systematic error or bias. If a trend develops, an investigation must be made as to the cause, corrections made, and the situation monitored. A trend is defined as six successive points going in the same direction, fourteen points in a row alternating up and down, or fifteen points in a row within (mean+1 $\sigma$ ) and (mean-1 $\sigma$ ).

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data must represent the sample analyzed regardless of the heterogeneity of the original sample matrix. Some samples may require analysis of multiple phases to obtain representative results. Data representativeness for a specific sample is affected by the validity of sample holding time, preservation, and lack of contamination. Representativeness is also used as a qualitative term evaluating if the data appropriately reflects the sample project site and parameters measured or studied.

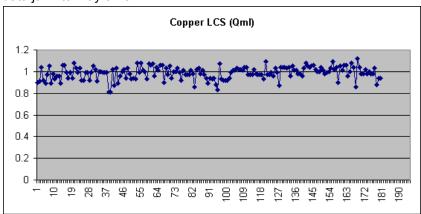
Comparability is the qualitative term that expresses the confidence that two data sets can contribute to a common analysis an interpolation. Comparability must be carefully evaluated to establish whether two data sets can be considered equivalent in regard to the measurement of a specific variable or groups of variables.

Completeness is a measure of the amount of valid data obtained from a measurement system expressed as a percentage of the number of valid measurements that should have been collected (i.e., measurements that were planned). Data may be considered not valid due to analytical problems or errors, sampling errors such as broken bottles or poor location, or other site-specific reasons.

Representativeness, comparability, and completeness are often best judged by the data end user. It may be necessary to adjust a sampling plan or re-sample to best measure site conditions.

<u>Uncertainty</u> is the sum of all errors expressed as a  $\pm$  value. Since it is impossible to know the value of all errors in an analytical procedure, the uncertainty is usually estimated. Given a big enough population, 95% of all measurements will fall within  $2\sigma$  of the mean (assuming a normal distribution). Uncertainty can then be calculated as  $\pm 2\sigma r$ , where  $\sigma r$  is the relative standard deviation ( $\sigma r = \sigma/X_{avg}$ ) and X is the value of the measurement. Since sample measurements are not generally carried out in statistically significant numbers of replicates, uncertainty is estimated from the LCS and LCSD data. If the population of values is small (<50) the student t value is used instead of 2. Uncertainty estimates are made when the control limits are calculated (twice/year). Following is an example of uncertainty calculation.

06/03/02 data jan1 to may 31 02



Uncertainty is Avg +/- 2σr 0.987056Avg 2σr is 0.117 0.057797StdDev L U Uncertainty is 0.99+/-0.12 0.115593wl 0.871 1.103 0.17339cl 0.814 1.16

More information on uncertainty can be found on the web and in the following documents:

Uncertainty and Dimensional Calibrations, Journal of Research of the National Institute of Standards and Technology, Volume 102, Number 6, November-December 1997.

Guidelines for Uncertainty Estimation, AIHA, Revision 0, November 28, 2001.

Measurement Uncertainty, UKAS, <a href="https://www.ukas.com/text\_only/information\_center/technical/technical\_uncertain.asp">www.ukas.com/text\_only/information\_center/technical/technical\_uncertain.asp</a>

EA-4/02, Expression of the Uncertainty of measurement in Calibration, European co-operation for Accreditation, December 1999.

If traceable calibration is not available or appropriate, comparison to a widely used standard may be used to demonstrate precision and accuracy. The standard and method used should be agreed upon by both client and lab prior to start of project. SOP for tests that have positive/negative or similar results give criteria for acceptance or rejection of QC batches.

### **Reporting Limits**

Assuring the validity of quantitative measurements at low concentrations is an extremely difficult technical problem. With regulatory action levels being pushed lower and lower, the validity of any given measurement becomes even more important. The consequences of false positive or false negative data can be significant.

Reporting Limits (RL) are established for each analyte in each method. RLs are based on Method Detection Limits (MDL), determined as described in 40 CFR 136. MDLs are the lowest concentrations above zero that we can identify and report as present with 99% confidence. Parameter MDLs are determined for each matrix or media tested. MDL studies are repeated each year or with each change in methodology or instrumentation. Reporting limits are set at a level above which we are confident that our laboratory can detect and quantify consistently. In general, the lowest calibration standard is used as a reporting limit. Reporting Limits are adjusted for sample dilution.

### 14. DATA REDUCTION, VALIDATION AND REPORTING

#### **Data Reduction and Validation**

The analyst enters results generated during the analysis of samples into the Laboratory Information Management System (LIMS). Each step of the data entry, validation, and approval is password protected to ensure data integrity and confidentiality. The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. Each analyst reviews the quality of his or her work to ensure that:

- Sample preparation information is correct and complete.
- Analysis information, including calibration and calibration verification, analyte identification, calculations (including manual calculations), and quantification, is correct and complete.
- The appropriate SOPs have been followed including the procedures for manual integration as detailed in Manual Integration SOP.
- Analytical results are correct and complete. (Occasionally it is necessary to change analytical results for valid reasons, such as calculation or data entry errors. If a change is necessary, the original results are never erased or obliterated with correction fluid. Data is changed by making a single line slash through the old result, and recording the new result, along with the initials of the person making the correction and the date of the correction.)
- Transcription of data is accurate.
- QC samples are within established control limits.
- Blanks are within appropriate QC limits.
- Special sample preparation and analytical requirements have been met.
- Documentation is complete (e.g., all anomalies in preparation and analysis have been documented; missed holding times are documented, manual integrations are indicated, etc.).

The analyst passes the data package for peer review. The peer review involves the same checks as the analyst and also includes:

- Calibration checks are adequate.
- Qualitative and Quantitative results and calculations are correct.
- Validation of the data package, both in the computer and by initialing raw data.

After initialing, the package is passed on to reporting. The report package is printed and assembled by the reporting clerk who checks the package for completeness. The project manger or designee then reviews the report package. This review shall provide a total overview of the data package, including sample receipt, to ensure its consistency and compliance with project-specific requirements. Calculations and data transfers to LIMs are checked against hard copy data. All errors noted shall be corrected and documented or qualified. Based on the errors noted, samples may need to be re-prepared and reanalyzed.

The QA department reviews at least ten percent of the data packages.. The QA review includes verifying the following:

- The sample checklist is completed.
- Report agrees with sample information found on the COC.
- Holding times have been met.
- QC results are complete, accurate and acceptable.
- Technical details are complete and reasonable.

In addition, calculations (manual and computer) are randomly checked for accuracy. Industrial Hygiene Badge calculations are checked at the rate of one per set. Records of such calculation validation are kept by the QA Officer/designee.

If no problems are found with the data package, the data package is approved by the Project Manager. or designee. The final report is then reviewed and signed by the QA Officer, Lab Manager or the Technical Manager, or Project Manager. Reports sent by electronic data transfer are first converted to PDF documents. Authorization of electronic signatures is given only by the Technical Manager, Laboratory Director, Environmental Services Director, Department Manager, or Quality Assurance Officer.

The completed report is copied and sent to the client along with a copy of the COC and the sample checklist. Figure 14-1 describes the flow of the data validation process.

The LIMS is backed-up daily by the designated personnel. The Gas Chromatography and Gas Chromatography/Mass Spectrometry files are not on the LIMS. These files are backed-up at least once per week to CD, by the chief chemist or supervisor responsible for performing the analyses.

### **Analytical Reporting**

The A & B Labs' reports are formatted in accordance with the specific requirements of the contracting agency or client. In general, the reports contain:

- Contracting Agency or Client
- Sampler
- Sample identification
- Date and time of sampling
- Date of report
- Date of analyses
- Parameter(s)
- Analytical Results
- Analyst
- Units of measure
- Methodology
- Sign-off
- Page number

The test report is sent to the client by fax, email (as PDF), and/or mail in a data package that contains:

- Signature page containing client and project information, and number of pages in the package.
- Sample ID-Lab ID cross-reference.
- Test report
- QC report.
- Chain-of-Custody.

Sample Condition Checklist.

Upon request, reports may be emailed as a spreadsheet to facilitate client record keeping.

When necessary for the interpretation of the test results or at the client's request, a statement on the estimated uncertainty of is given as a  $\pm$  statement in the results or in the case narrative..

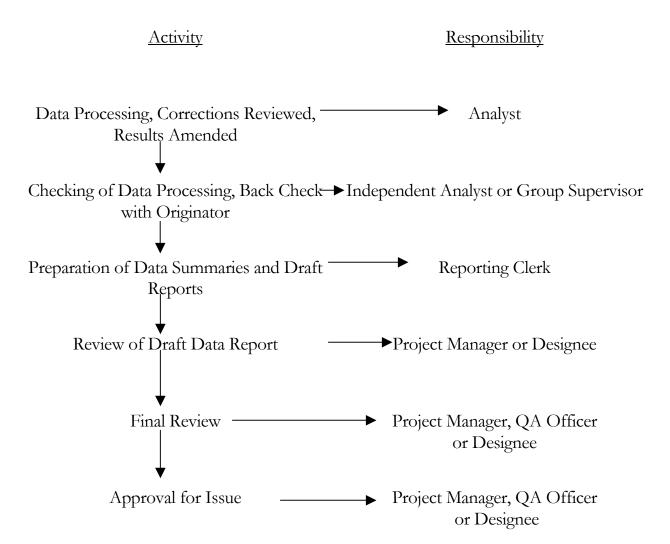
If additional analysis is requested, a supplemental report is issued. The cover sheet and fax cover are clearly marked as amended or additional analyses. If for some reason an error is found in a report after it has been sent to a client, the client is notified by telephone and a new report is issued. The new report is labeled as a correction.

### **Client Confidentiality**

A & B Labs is concerned with the protection of client confidentiality and proprietary rights at all times. Analytical reports and information relating to the testing and samples are released only to the client or their representative. Analytical data may be released to someone else, but only with written permission from the client. Authorized personnel who have access to client information may not divulge it in whole or part to any person or organization other than the client, without written authorization from the client.

Additionally, personnel may not alter any phase of sample receipt, preparation, analysis or reporting in any manner that may be determined to be falsification of the records. Any employee discovered falsifying records will be dismissed immediately, and may be subjected to civil and/or criminal charges.

Figure 14-1
Data Validation Process



### 15. PERFORMANCE AND SYSTEM AUDITS

A system audit is a review of the laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff and procedures in place to generate acceptable data.

A performance audit verifies the ability of the laboratory to correctly identify and quantify compounds in blind check samples.

A & B Labs participates in the analyses of Water Supply (WS) and Water Pollution (WP) Performance Evaluation (PE) studies from Resource Technology Corporation (RTC). A & B Labs has NELAC certification with the State of Florida and secondary certification with the State of Louisiana based on the results of the RTC PE studies and other certification requirements. A & B Labs also participates in the AIHA PAT and ELPAT PE studies quarterly. The results of all PE samples are evaluated by the QA Officer.

The QA Officer submits blind check samples to the laboratory periodically. The frequency and type of samples sent is based on problem areas identified by evaluation of Performance Evaluation results, or the desire to check performances prior to the analysis of samples in general.

External system audits are performed by the NELAC and AIHA on a three year schedule.

In addition to the external audits conducted by certifying or regulating agencies, A & B Labs conducts the following internal audits:

- The QA Officer performs a system audit on a yearly basis.
- The QA officer performs special audits whenever a problem is suspected or there is an analyst complaint.
- Any problems found in an internal audit generates a corrective action report. Deficiencies are corrected or resolved in a timely manner.
- If it is determined that deficiencies found in an audit affect the validity of test results, clients shall be notified in writing of the deficiency, corrective action, and degree to which their results are affected.
- An annual management review is conducted, which reviews the quality

system to ensure continued suitability.

### Management reviews should include:

- Suitability of policies and operations procedures.
   Review of policy and operation changes made during the year and their effectiveness
- Management reports
   Review of minutes of management meetings
- Summarized internal audit results
  Review of audits and deficiency corrections made during the year
- Corrective and preventive actions
   Review of corrective and preventive action reports
- Assessment of external auditors
   Review of customer and agency external audits
- Results of proficiency tests and inter-laboratory comparisons
   Review of results from proficiency testing and inter-laboratory comparisons
- Changes in volume or type of testing
   Review of department volumes and changes in types of testing
- Client complaints and communications
   Review of client complaints (and corrective actions, if any) and communications recorded with sample folders
- Staffing resources and training requirements
   Review of overtime hours and employee training

Findings from management reviews and the actions that arise from them shall be recorded. Management shall ensure that those actions are carried out within an appropriate and agreed timescale. Results of internal audits and management review shall be shared with laboratory personnel in the monthly safety/QA meetings. Records of audits and reviews, corrective and preventive actions are retained for a minimum of five (5) years.

# 16. NONCONFORMANCE AND CORRECTIVE ACTION

#### Nonconformance

A nonconformance is a deficiency in documentation or procedure sufficient to render the quality of an item unacceptable or indeterminate, or any event that is outside documented procedures for laboratory operation.

Nonconformance may include the following:

- Failure of an instrument to work properly.
- Sample receiving documentation not correct.
- Sample condition on receipt not acceptable.
- Sample holding time exceeded.
- Sample storage conditions outside criteria.
- Incorrect sample preparation/analysis procedures.
- Calibration requirements not met.
- Data validation errors.
- Relative standard deviation for response factors greater than accepted limits.
- QC data are outside the limits for precision and accuracy.
- CCV standards do not pass requirements.
- Blanks or LCS contain contaminants.
- Undesirable trends are detected in spike recoveries or RPD between duplicates.

- Deficiencies are detected by QA during internal or external audits or from the results of PE samples.
- Inquiries concerning data quality are received from client or other analysts involved with the production of the data.

### **Root Cause Investigation**

The resolution of nonconformances and deficiencies requires a root cause analysis. The steps in a root cause investigation are different depending on the type of nonconformance, the complexity of the problem, and the range of impact. The chart below gives the steps along which samples and data flow. The starting point of an investigation depends on the nature of the nonconformance.

Work Flow Personnel Sample Method Controls Data Policies Validation Log-in Preparation Sample Trail Procedures Handling/Storage Logbook entries Routing Reagents Training Instrumentation Control Charts Calculations Storage Software Final Report

Figure 16-1 Work Flow

The first step of corrective action is documentation of the problem. A nonconformance/corrective action form is initiated by the employee who discovers the problem. The following check list is used to direct the flow of investigation.

#### Personnel

- Interview employees involved in the work associated with the affected samples.
- Is the level of training and experience of the involved staff members adequate?

# Sample

- Were all minimum sample acceptance criteria met? Was there anything unusual about the sample noted upon receipt?
- Are the log-in records complete and correct?
- Was the sample routed to the proper analysts?
- Were the samples stored properly upon receipt and up to the time of analysis?

#### Method

- Was the SOP followed? Are there any deficiencies in the SOP?
- Review the method validation records. Have any of the established method parameters changed over time?
- Check preparation of standards, reagents, and any test supplies that might have a critical impact on the test results.
- Were the instrumental calibrations done properly? Review past calibrations for any major changes. Review the instrument logbook records.

#### Controls

- Critically review all aspects of the QC data itself.
- Were the QC samples prepared properly?
- Were the control materials properly stored? Had any of the control materials expired?
- Review the data transfer to control charts. Check the formulae for any automatic calculations.

#### Data

• Review the raw data carefully. There may be transcription or transposition

errors.

- Check for gaps in the sample trail. Was the sample under proper custody at all times?
- Check logbook entries.
- Recheck calculations.
- Insure the integrity of the formulae used for computer calculation steps.
- Is all the information provided in the final report accurate? Are there any inconsistencies between the final report and the analytical history traced via the investigation?

#### **Corrective Action**

Once the root cause for any nonconformance has been established, corrective actions must take place. Resumption of work on a non-conforming QC batch is authorized by the Department Manager. Any nonconformance which affects the accuracy of analytical data requires a reanalysis of the samples involved. If because of holding times or sample volume this is not possible, a resample is requested from the client. Once resolved, any client whose work may have been affected receives a letter explaining the problem, the cause, and our subsequent corrective action taken to avoid future occurrences of this type. Full documentation of the corrective action procedure is filed with the project records and a copy is kept in the QA Office. Corrective actions are tracked by referring to the corrective action file in the QA office. See figure 16-1 for an example corrective action form.

#### **Preventative Action**

Recurring corrective actions (2) are discussed in management meetings to determine possible preventative actions. Preventative action may include but are not limited to extra training for employees, instrument service, or changes to SOP's. An important part of preventative action is the follow up of the procedures implemented in corrective actions. Checks are made within thirty days of a corrective action to determine its effectiveness.

Preventative action is used proactively to guard against the need for corrective

actions. Management meeting of all supervisors and managers are held every two weeks to discuss any problems or suggestions for improvements. QA reports to the management are given at this time. Monthly Safety/QA meetings are held with the entire staff to inform employees of any changes decided on in management meetings and results of PT studies. Employees are encouraged to take the floor and air any problems or suggestions.

# Figure 16-2 Sample Corrective Action Report

| Complaint/Nonconformance/Corrective Actions  |            |
|--|------------|
| Client or contact  |            |
| Complaint rec'd by:  | Passed to: |
| Date:  | Lab ID:    |
| Problem:   |            |
|  |            |
|  |            |
| Steps Used to Determine Cause  |            |
| Steps Used to Determine Cause  |            |
|  |            |
|  |            |
|  |            |
|  |            |
|  |            |
|  |            |
| Corrective Actions   |            |
|  |            |
|  |            |
|  |            |
|  |            |
|  |            |
| The state of the s |            |
| Preventive Action  |            |
|  |            |
|  |            |
| Analyst  |            |
| QA Officer/designee  |            |
| Management   |            |

#### 17. Client Communications

Project bids and contracts are reviewed by the Technical Manager, Lab Director, QA Officer, Environmental Services Director, Microbiology Services Director, Chief Chemist, and/or Project Manager who determine if A & B has the instrumentation, experience, and other resources necessary to complete the analytical requirements of the client. The review may encompass results of earlier analytical testing or proficiency testing and/or the running of trial environment programs using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc. The appropriate analytical method is selected which is capable of meeting the clients requirements. Any differences between request or tender and the contract shall be resolved before any work commences. Each contract shall be acceptable both to the laboratory and the client.

Contracts include not only long-term commitment to a given quoted project but also daily promises made in the form of accepting samples and chains-of custody.

Ambiguous analytical requests made on a chain-of-custody shall be resolved before the sample is accepted for analysis. The Customer Service Representative or Project Manager is responsible for contacting the customer and clarifying the request. If customer response is slow, the sample is logged in and taken to the cooler. A written, faxed request for analysis is preferred over a verbal response. If the question is one of poor penmanship, a verbal response is adequate. All discussions with a client about a sample or project must be written in either a phone log or on the project folder.

If the analysis request is for work not done at A & B, the customer is informed by phone or fax as soon as is practicable. If the customer desires that the work be subcontracted, the samples are sent to a laboratory of their choice. Any subcontracted work is sent to a laboratory with valid accreditation for the specific analytical parameter. Asbestos samples are sent to a NVLAP accredited lab. All data from a subcontracted lab are reported as being tested by outside laboratory. A list of currently acceptable subcontracting entities is kept.

Any addition or amendment to the analysis request shall be obtained in writing by fax whenever possible. The Project Manager or Customer Service Representative shall inform the data log in employee of the additional analysis. Each analyst

concerned shall also be notified. The holding times of the additional analysis must be carefully checked before accepting the work. Any report made of added analyses shall be labeled additional or supplemental to the original report.

Complaints from clients are recorded on the project folder concerned. Any corrective action report generated as a result of the complaint is kept in the project folder or in the QA office. The QA officer to determine effectiveness reviews the corrective action reports monthly.

Communications between A & B and its clients must be maintained during any project. Any changes in schedule or instrument status or scope of work that might affect a project are to be communicated to the client immediately. All communications with the client concerning a specific in house project are recorded on the project folder. Any changes in scope of analytical work requested by the client are communicated to the analyst immediately. Clients are encouraged to tour the lab to ascertain facilities and capabilities in relation to work performed. Confidentiality to other clients must be maintained.

Confidentiality of client information and data is important to A & B Labs. A & B Labs is concerned with the protection of client confidentiality and proprietary rights at all times. Analytical reports and information relating to the testing and samples are released only to the client or their representative. Analytical data may be released to someone else, but only with permission from the client. Authorized personnel who have access to client information may not divulge it in whole or part to any person or organization other than the client, without written authorization from the client.

# 18. QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. The QA Officer must report the following to the President or Technical Manager:

- The results of internal systems audits including any corrective actions taken.
- Performance evaluation scores and commentaries.
- Results of site visits and audits by regulatory agencies and clients.
- Performance on major contracts.
- Problems encountered and corrective actions taken.
- Holding time violations.
- Comments and recommendations.

#### 19. LABORATORY DOCUMENTATION

#### Records

In order to achieve traceability of measurements complete and accurate documentation of sample receipt, preparation, analysis procedure, and report information is a necessary part of the QA program. The records for each test shall contain sufficient information to facilitate reconstruction of the quantification process, to enable the test to be repeated under conditions as close as possible to the original, and, if possible, to identify factors affecting the uncertainty. All records are maintained for a minimum of five (5) years. At the end 5 years the records are shredded and disposed of.

No unauthorized person has access to laboratory documents. All client reports, chains of custody, and backup copies of the data are kept in a secure area accessible only by authorized personnel.

All archived documents are kept in a document storage area with controlled access.

All laboratory notebooks have control numbers, and archived notebooks are filed in the QA Office for one year then moved to the document storage area.

The following describes the documentation used in A & B Labs.

#### Field Records

It is the responsibility of the field supervisor to assure that all appropriate paperwork, specifically, the Chain-of-Custody form is initiated. Field records are maintained in a waterproof notebook and all entries are made with waterproof ink, to aid in verification of sample integrity. Calibrations and data generated in the field are part of laboratory documentation and shall be turned in to the Environmental Director to become part of laboratory records.

# Laboratory Notebooks

Laboratory notebooks and/or computerized records are maintained to document instrument maintenance and standard preparation and concentration.

Laboratory/computer notebooks are used to document information from routine laboratory operations, including sample preparation and analysis. These notebooks are also used to ensure that the information is recorded in a complete and organized manner and the analysis can be reconstructed. No original calibration records or other data shall be removed from the laboratory. Copies may be made for purposes of field calibration or data analysis on home computers only with approval of a supervisor.

#### **Control Charts**

A & B Labs uses control charts to visually track the precision and accuracy data. These control charts are used to identify trends in the analyses which may indicate a problem with the analytical procedure, and to monitor the reliability of the data. Without an indication of reliability, it is difficult to support analytical results and the decisions based on them. Whenever an adverse trend is detected corrective action is performed.

#### **SOPs**

Details of analytical, QC and safety protocols are contained in SOPs. SOPs are documents that contain detailed proprietary information on how to perform a laboratory procedure.

All SOPs must be approved by the QA Officer and Management. The distribution of current SOPs and archiving of outdated ones is controlled through the QA Office. SOPs are revised whenever there is a method revision. Whenever the QA Office receives a method revision, the supervisor and the analyst who performs the analysis are informed. The analyst familiarizes him or herself with the revisions, validates the changes with analytical data, and revises the SOP accordingly. The revised SOP is then submitted to the QA Officer for approval. After the revisions are discussed and approved, the SOP is assigned an identification number, signed and dated. Hand-written SOP identification numbers, revision numbers and effective dates are acceptable, only if they have been assigned by the QA Officer and the original remains in the QA Office. SOPs are logged in to the BTLIMS and made available for viewing on the local network.

Because of the detailed nature of SOPs, A & B considers them to be proprietary documents.

The formats for A & B SOPs are given in Appendix II.

### **Project Files**

The project file consists of copies of reports, chain of custody, sample checklist and any correspondence. The project file also includes sample data and QC data on report sheets. Raw data is also included (i.e., chromatograms).

In the event that the laboratory transfers ownership or goes out of business, clients will be given the option of storing their own records or the records will be placed in a storage facility for a period of 5 years by A&B management.

### **Computer Records**

The LIMS database is backed up to a hard drive other than the server every day. The LIMS is backed up to CD once a month. The CDs are accessible only through the laboratory director. The LIMS data base is password protected to prevent unauthorized access.

Computer records generated by the GCs and GC/MSs are backed up daily to a password protected external hard drive. Hard copies of the data generated by GC runs are included in each project folder. Asbestos data is backed up to CD once per month.

Calculations made by computers are validated manually when the form or spreadsheet is set up. Formulas are then locked to prevent inadvertent change. Data transfers to LIMS are checked against hard copy data in the reporting process.

## Laboratory Documents and Forms

Forms are needed in the laboratory to organize information and data. Forms are issued after discussion with analysts and supervisors. New or revised forms are reviewed and approved by management. After approval by Lab Director and QA Officer, the new form is assigned an inventory or other unique identification number. Forms are logged in to a spreadsheet (master document list) with version number and date of approval. Revised forms are distributed to appropriate areas.

Formatting for forms shall include a unique identifier, date of issue or revision, and for multiple page documents, page numbers, total number of pages, or a mark identifying the end of the document. Single page forms used to organize data before reporting, shall be initialed by the analyst when completed.

Authorized documents shall be available where needed, and reviewed at least yearly by the supervisor or manager concerned. Invalid documents shall be removed promptly and marked as obsolete. No form can be altered without approval from QA Officer or Lab Director. Obsolete documents retained for either legal or knowledge preservation purposes are clearly marked to indicate status.

Changes to documents shall be reviewed and approved for use by management. Where practicable, the altered or new text shall be identified in the document or the appropriate attachments. Computerized document changes must be controlled and managed. Reports are saved as PDF files. Whenever changes are necessary, the new PDF file is saved as a corrected or amended report.

Once a form is used, it becomes a record and is stored as already discussed. SOPs, software, instruments manuals, and reference books are also considered to be documents. As such they are logged in to the master document list as an aid to tracking version and use. Old versions are archived into storage.

The Master Document List is continually updated by the QA Officer, or representative, as forms, SOPs, and other documents are revised and added. The forms on the Master List are divided by use and purpose. The area of distribution and date of revision or initiation is listed. Computer Software is treated as any other form, and listed by purpose. Any software that is used to calculate analytical results or QC parameters must be validated at the time the form is put into use. The date of validation is entered into the Master List.

Some of the forms used at A & B follow:

Sample handling forms:

Chain-of-custody general Chain-of-custody Industrial Hygiene Chain-of-custody Bulk Asbestos Chain-of-custody subcontract Sample condition checklist etc.

#### Bench Sheets:

Metals Digestion

Mercury Digestion

Mercury Analysis

Group Analyses (such as drinking water metals and TCLP metals)

Wet Lab bench sheets (such as TKN, NH<sub>3</sub>, BOD, and etc.)

ICP run log

ICP run data

GC tune

GC cal check

GC data

GC data summary

IC run data

Asbestos PCM Form

Asbestos PLM Form

etc.

# Quality Check forms:

Data quality checklist

Balance calibration check

Refrigerator check

Thermometer check

Production log

Nonconformance/corrective action

GC/MS SPCC and CCC

etc.

### Report forms:

Cover sheet

IH cover sheet

Analytical report

QC report

etc.

#### Procurement forms:

Purchase request

Purchase order

Vendor evaluation Reagent and standard receipt etc.

#### Log books (written information in bound books):

Samples received Instrument maintenance Standard preparation etc.

### Computer Software:

EZ Chrom
EnvironQuant
PeakNet
WinLab32
AT Sample Master
Windows 2000, XP, NT
MS Office
Adobe Acrobat
etc.

#### Reference Books

EPA, Methods for Chemical Analysis of Water and Wastes, March 1983
APHA, Standard Methods for the Examination of Water and Wastewater, 20<sup>th</sup> Edition, 1998
EPA, Test Methods for Evaluating Solid Waste, SW-846, 1996 etc.

#### **Equipment Manuals**

HP 5890 Series II Service Manual Tekmar 2016/2032 Users Guide

#### 20. LABORATORY CONSUMABLES

Laboratory supplies and consumables are ordered by the Office Manager. Whenever supplies are needed the analyst fills out a requisition in the ICM program stating the type, quality, amount and the date the supplies are needed. Quality of reagents ordered shall meet method specified requirements or be analytical grade. When the supplies arrive the receiving clerk checks the supplies against the packing slip and the requisition. Any discrepancies are reported to the Office Manager, who settles the discrepancies with the supplier. All packing slips are given to the Office Manager.

#### Reference Standards

Reference standards, such as the S-class weights used for balance checks and the traceable thermometer used for thermometer checks, shall be purchased precalibrated from an ISO certified vendor. Calibration shall be traceable to national standards of measurement. Calibration shall be checked yearly. Reference standards shall be used only for calibration unless it can be shown that their use does not invalidate their performance as reference standards.

### Reference Materials and Reagents

All reference materials and reagents are assigned a log number (LT#) by the ICM program upon receipt. The ICM logbook record includes: the date received, name of standard or reagent, lot number, expiration date, vendor and the logger's initials. The log number (LT#) is used to identify standard or reagent in lab notebooks and prep books providing traceability to vendor lot. Standards and reagents are distributed to the analyst specified in the ICM requisition form. The analyst is responsible for storing the standards or reagents as recommended by vendor, method, or agency.

Reagent and standard preparation is recorded in reagent prep logs. Records include traceability to purchased stocks, method of preparation, date prepared, expiration date, and analyst's initials. Each prepared reagent and standard is assigned and labeled with a unique identifier (LLT#) and an expiration and prep date.

Suppliers of reference materials must have a quality system registered to one of

the ISO 9000 standards. Vendors are evaluated by the laboratory personnel who use them. Records of evaluation and approval of suppliers are retained for five (5) years.

No reference materials, reagents or chemicals may be used until they have been verified and found acceptable (acceptable blank, calibration, and/or positive/negative controls), or in agreement with the Certificate of Analysis.

No reference materials, reagents or chemicals may be used after the expiration date, whether the expiration date is listed, or determined based on method demands. An exception to this policy can be made only if re-certification of the materials has been performed and a new expiration date has been assigned.

All expired reference materials, reagents or chemicals are disposed in the proper manner.

#### Glassware and Volumetric Apparatus

Only Class A quality glassware is used when the highest degree of accuracy is needed. Pipets and micropipets are calibrated by mass. Disposable measuring apparatus are calibrated at the rate of one per lot.

## Reagent Water

Records are kept of the quality of the reagent water used in the lab. A daily check is made of the conductivity (acceptance criteria <2µmhos). A monthly check is made of Chlorine (acceptance criteria <0.1mg/L) and pH (acceptance criteria 5 to 8).

#### 21. PREVENTIVE MAINTENANCE

Computers and automated equipment are maintained to ensure proper functioning and are provided with the environmental and operating conditions necessary to maintain the integrity of test and calibration data. To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument.

Laboratory instruments are periodically subjected to the preventive maintenance tasks as outlined by the manufacturers. The manufacturers equipment manual is retained and consulted for procedures for safe handling, transport, storage, use and planned maintenance of equipment to ensure proper functioning and in order to prevent contamination or deterioration. An inventory of operator replaceable parts is maintained by A & B Labs for each analytical instrument. Equipment that has been subjected to overloading, gives defective results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labeled or marked as being out of service until it has been repaired and shown by calibration or test to perform correctly. If repairs are necessary, they are done by either trained staff or trained service engineers employed by the instrument manufacturer.

Preventive maintenance for GCs includes regular replacement of septa, cleaning or replacement of injector liner, and replacement of ferrules. MS maintenance includes checks on gas pressure, tunes, and vacuum status.

A & B Labs also maintains an inventory of parts for all sampling equipment and performs preventive maintenance on them prior to any sampling event.

The laboratory also maintains detailed logbooks documenting the preventive maintenance performed on each analytical instrument, and the frequency at which the maintenance is performed.

## 22 Advertising

A & B Laboratories participates in a number of certification programs. Each accrediting body has specific rules for how their name and logo may be displayed. These rules are on file in the QA office or may be found on the web at each individual web site. At a minimum, the laboratory should make available their scope of accreditation to all clients. All advertisements containing the name of an accrediting body should also have the Lab ID Code or certificate number for A & B Labs.

#### 23 REFERENCE

Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019, March 1979

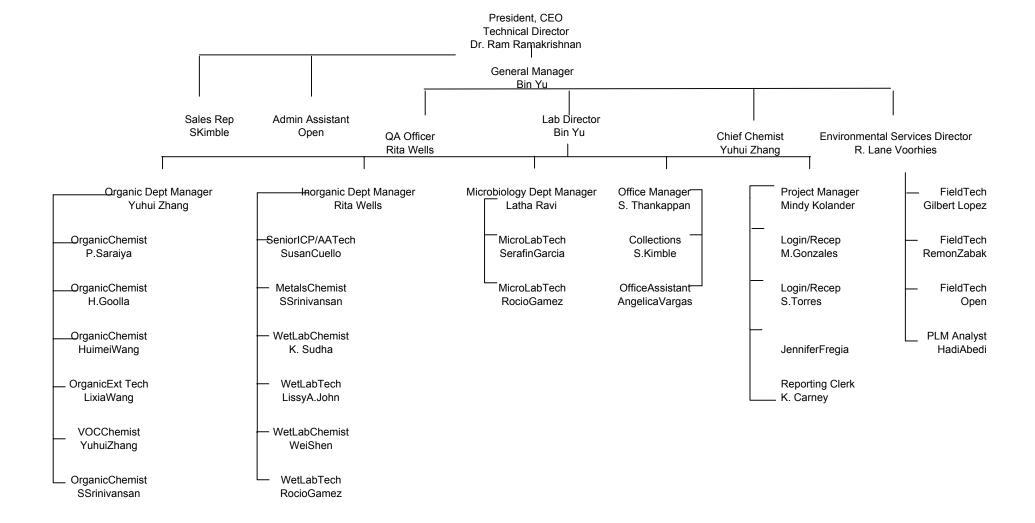
Standard Methods for the Examination of Water and Wastewater, APHA, 20<sup>th</sup> Edition, 1998

Laboratory Quality Assurance Programs Policies, AIHA, February 2002

Quality Systems, National Environmental Laboratory Accreditation Conference, November 4, 2002.

Quality Systems, National Environmental Laboratory Accreditation Conference, June 5, 2003.

# APPENDIX I ORGANIZATIONAL CHART



### APPENDIX II

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR SOLID WASTE AND WASTEWATER

Table II-Required Containers, Preservation Techniques, and Holding Times

| Table II-Required Containers, Pre  | Container <sup>1</sup> | Preservation <sup>3,5</sup>                                      | Maximum holding time <sup>4</sup> |
|--|------------------------|--|-----------------------------------|
| Table 1A-Bacteria Tests:   |                        |  |                                   |
| 1.4 Coliform, Fecal and total  | P, G                   | Cool, 4 °C, 0.888% Na <sub>2</sub> S <sub>2</sub> o <sub>3</sub> | 6 hours                           |
| 5 Fecal streptococci   | P, G                   | Cool, 4 °C, 0.888% $Na_2S_2o_3$                                  | 6 hours                           |
| Table 1A-Aquatic Toxicity Tests:   |                        |  |                                   |
| 6-10 Toxicity, Acute and Chronic   | P, G                   | Cool, 4 °C <sup>36</sup>   | 6 hours                           |
| Table 1B-Inorganic Tests:  |                        |  |                                   |
| 1. Acidity   | P, G                   | Cool, 4 °C   | 14 days                           |
| 2. Alkalinity  | P, G                   | do   | do                                |
| 4. Ammonia   | P, G                   | Cool, 4 °C, H <sub>2</sub> SO <sub>4</sub> to pH<2               | 28 days                           |
| 9. Biochemical oxygen demand   | P, G                   | Cool, 4 °C   | 48 hours                          |
| 10. Boron  | P, PFTE, or Quartz.    | HNO <sub>s</sub> to pH<2   | 6 months                          |
| 11.Bromide   | P,G                    |  | 28 days                           |
| 14.Biochemical oxygen demand, carbonaceous   | P,G                    | Cool, 4 °C   | 48 hours                          |
| 15. Chemical oxygen demand   | P, G                   | Cool, 4 °C, H2SO4 to pH<2  | 28 days                           |
| 16. Chloride   | P, G                   | None required  | do                                |
| 17. Chlorine, total residual   | P, G                   | do   | Analyze immediately.              |
| 21. Color  | P, G                   | Cool, 4 °C   | 48 hours                          |
| 2324. Cyanide, total and amenable to chlorination.   | P, G                   | Cool, 4 °C, NaOH to pH.12, 0.6g ascorbic acid.                   | 14 days. <sup>6</sup>             |
| 25. Fluoride   | P, G                   | None required  | 28 days                           |
| 27. Hardness   | P, G                   | $\mathrm{HNO_{3}}$ to pH<2, $\mathrm{H_{2}SO_{3}}$ to pH>2       | 6 months                          |
| 28. Hydrogen ion (pH)  | P, G                   | None required  | Analyze immediately               |
| 31,43. Kjekhhi and organic nitrogen  | P, G                   | Cool, 4 °C, H <sub>2</sub> SO <sub>4</sub> to pH<2               | 28 days                           |
| Metals:  |                        |  |                                   |
| 18. Chromium VI  | P, G                   | Cool, 4 °C   | 24 hours                          |
| 35. Mercury  | P, G                   | HNO <sub>5</sub> to pH-2   | 6 months                          |
| 3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 51, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI and meroury | P, G                   | do   | 6 months                          |
| 38. Nitrate  | P,G                    | Cool, 4 °C   | 48 hours                          |
| 39. Nitrate-nitrite  | P, G                   | Cool, 4 °C, H <sub>2</sub> SO <sub>4</sub> to pH<2               | 28 days                           |
| 40. Nitrite  | P, G                   | Cool, 4 °C   | 48 hours                          |
| 41. Oil and grease   | G                      | Cool to 4 °C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2      | 28 days                           |
| 42. Organic Carbon   | P, G                   | Cool to 4 °C, HCl or $H_2SO_4$ or $H_3PO_4$ pH-2                 | 28 days                           |
| 44. Orthophosphate   | P,G                    | Filter immediately Cool, 4 °C                                    | 48 hous                           |
| 46. Oxygen, Dissolved Probe  | G, Bottle and top.     | None required  | Analyze immediately.              |
| 47. Winkler  | do                     | Fix on site and store in dark                                    | 8 hours                           |

| 48. Phenols  | G only                     | Cool, 4 °C, $\rm H_2SO_3$ to pH<2                                | 28 hours   |
|--|----------------------------|--|--|
| 49. Phosphorus (elemental)   | G                          | Cool, 4 °C   | 48 hours   |
| 50. Phosphorus, total  | P,G                        | Cool, 4 °C, $\rm H_2SO_3$ to pH<2                                | 28 days  |
| 53. Residue, total   | P, G                       | Cool, 4 °C   | 7 days   |
| 54. Residue, Filterable  | P,G                        | do   | 7 days   |
| 55. Residue, Nonfilterable (TSS)   | P, G                       | do   | 7 days   |
| 56. Residue, Settleable  | P,G                        | do   | 48 hours   |
| 57. Residue, Volatile  | P, G                       | do   | 7 days   |
| 61. Silica   | P, PFTE, or Quatz          | Cool, 4 °C   | 28 days  |
| 64. Specific conductance   | P,G                        | do   | do   |
| 65. Sulfate  | P, G                       | do   | do   |
| 66. Sulfide  | P, G                       | Cool, 4 °C, add zinc acetate plus<br>sodium hydroxide to pH>9.   | 7 days   |
| 67. Sulfite  | P, G                       | None required  | Analyze Immediately                                |
| 68. Surfactants  | P,G                        | Cool, 4 °C   | 48 hours   |
| 69. Temperature  | P, G                       | None required  | Amilyze  |
| 73. Turbidity  | P,G                        | Cool, 4 °C   | 48 hours   |
| Table 1C-Organic Tests:  |                            |  |  |
| 13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56,<br>66, 88, 89, 92-95, 97. Purgeable<br>Halocarbons.   | G, Teflon-lined<br>septum. | Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> o <sub>3</sub> | 14 days  |
| 6, 57, 90. Purgeable Aromatic Hydrocarbons   | do                         | Cool, 4 °C, 0.008% Na <sub>0</sub> S <sub>2</sub> o <sub>3</sub> | do   |
| 3, 4, Acrolein and acrylonitrile   | do                         | Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> o <sub>3</sub> | . do   |
| 23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96.<br>Phenols <sup>11</sup>                                 | G, teflon-lined cap.       | Cool, 4 °C, 0.008% Na <sub>2</sub> S <sub>2</sub> o <sub>3</sub> | 7 days until extraction, 40 days after extraction  |
| 7, 38. Benzidines <sup>11</sup>  | do                         | do   | 7 days until extraction. <sup>13</sup>             |
| 14, 17, 48, 56-52. Phihalate exters <sup>11</sup>  | do                         | Cool, 4 °C   | 7 days until extraction, 40 days after extraction. |
| 72-74.Nitrosamines <sup>11,14</sup>  | do                         | Cool, 4 °C, store in dark, 0.008% Na_2S_2o_3                     | do   |
| 76-82. PCBs <sup>11</sup> acrylonitrile  | do                         | Cool, 4 °C   | do   |
| 54,55,65,69. Nitroaromatics and isophorone <sup>11</sup>   | do                         | Cool, 4 °C, 0.008% $Na_2S_2o_3$                                  | do   |
| 1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Poly<br>nuclear aromatic hydrocarbons <sup>11</sup> . | do                         | do   | do   |
| 15, 16, 21, 31, 75. Haloesthers <sup>21</sup>  | do                         | Cool, 4 °C, 0.008% Na <sub>3</sub> S <sub>2</sub> o <sub>3</sub> | do   |
| 29,35-37,60-63,91. Chlorinated hydrocarbons <sup>11</sup> .  | do                         | Cool, 4 °C   | do   |
| 87. TCDD <sup>II</sup>   | do                         | Cool, 4 °C, 0.008% $Na_2S_2o_3$                                  | do   |
| Table 1D-Pesticides Tests:   |                            |  |  |
| 1-70. Pesticides <sup>11</sup>   | do                         | Cool, 4 °C, pH 5-913   | do   |
| Table 1E-Radiological Tests:   |                            |  |  |
| 1-5. Alpha, beta and radium  | P,G                        | HNO <sub>3</sub> to pH<2   | 6 months   |
| Table II Notes:  |                            |  |  |

Table II Notes:

- <sup>1</sup> Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (poly propylene or other autoclavable plastic).

  <sup>2</sup> Sample preservation should be preformed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of
- collection. When an automated sampler makes it impossible to preserve each aliquot.

  SWhen a sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulation (49 CFR part 172). The person offering such material for transport is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Material Transport Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochleric acid (HCI) in water solutions at concentrations of 0.64% by weight or less(pH about 1.96 or greater); Nitric acid (HNO<sub>2</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
- Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or mornicaring laboratory, has data on file to show that the specific tes of samples under study, the analytes are stable for the longer time and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hole the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

  <sup>5</sup> Should only be used in the presence of residual chlorine.
- Maximum holding time is 24 hours when chloride is present. Optionally all samples maybe tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot is obtained. The sample is filtered and then NaOH is added to pH 12.

  - 24 is added to pH 12
    Samples should be filtered immediately on-site before adding preservative for dissolved metals.

    Samples should be filtered immediately on-site before adding preservative for dissolved metals.

    Guidance applies to samples to be analyzed by GC LC, or GC/MS for specific compounds.

    Sample receiving no pH adjustment must be analyzed with in seven days of sampling.

    The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- The per adjustment is not required in account with no commission, studies of account feet and the extracted early test of concern fall within a single chemical category, the specified preservative ans maximum holding times should be observed for optimum safeguard of the same integrity. When the analyses of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sociaum thiosulfate, storing in the dark, and adjusting the pH to 6-9, samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in foot note 5 (re the requirements for thiosalfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).
  - <sup>12</sup> If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine
- DExtract may be stored up to 7 days before analysis, if storage is conducted under an inert (exichnt-free) atmosphere.

  For the analysis of dipherylnitrosamine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> and adjust the pH to 7-10 with NaOH within 24 hours of sampling.

  The pH adjustment may be preformed upon receipt at the laboratory and may be omitted if the sample are extracted within 72 hours of collection. For the analysis of
- Sufficient ice should be placed with the samples in the shipping container to ensure that the ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive it is necessary to immediately measure the temperature of the samples and confirm that the 4°C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples are not reduced because of the increased holding temperature.

## **APPENDIX III**

FORMAT FOR ANALYTICAL STANDARD OPERATING PROCEDURES (SOPs)

Subject or Title: Page n of n

**Title** Method

SOP No: Version No. Effective Date: XXXXX X Month Year

Supercedes Revision No. X

- 1. Scope and Application
  - 1.1 Analytes
  - 1.2 Reporting range
  - 1.3 Applicable matrices
  - 1.4 Approximate analytical time (i.e., 5 minutes, hours)
- 2. Method Summary
  - 2.1 Generic description of method and chemistry behind it (i.e., extract with solvent, convert to methyl ester, analyze by electron-capture Gas Chromatography)
- 3. Comments
  - 3.1 Interferences
  - 3.2 Helpful hints
- 4. Safety Issues
- 5. Sample Collection, Preservation, Containers, and Holding Times
- 6. Apparatus
- 7. Reagents and Standards
- 8. Sample preparation
- 9. Procedure (detailed step-by-step)
  - 9.1 Calibration
  - 9.2 Sample Analysis
- 10. Calculations
- 11. QA/QC Requirements
  - 11.1 QC samples
  - 11.2 Acceptance criteria (precision and accuracy)
  - 11.3 Corrective action required
- 12. Reporting

|             |              |   | Standard<br>Operating       |
|-------------|--------------|---|-----------------------------|
|             |              |   | Procedure                   |
| Subje       | ct or Title  | ):<br>-   | Page n of n                 |
|             |              | <b>Title</b><br>Method                                    |                             |
| SOP<br>XXXX |              | Version No.<br>X  | Effective Date:  Month Year |
| Supe        | rcedes R     | evision No. X   |                             |
|             | 12.1<br>12.2 | Reporting units Reporting limits                          |                             |
| 13.         | Refere       | ences   |                             |
|             | 13.1<br>13.2 | Method source Deviations from source method and rationale |                             |
| Mana        | gement A     | Approval  |                             |
| QA A        | pproval      |   |                             |

# APPENDIX IV ANALYST TRAINING CHECKLIST

## Training Record

| Employee:   |  |                   |                    |
|---|--|-------------------|--------------------|
| I have read, understood, and am us  | ing the latest versi<br>Initials           | on of:<br>Date    |                    |
| The Quality Assurance Manu<br>The Health and Safety Manua   | al<br>al                                   |                   |                    |
| I have read and been trained in the   | following methods<br>Initials <sup>*</sup> | and SOPs:<br>Date | Trainer<br>        |
|   |  |                   |                    |
|   |  |                   |                    |
|   |  |                   |                    |
|   |  |                   |                    |
|   |  |                   |                    |
| *Initials here indicate that training ha<br>reporting, quality control, safety, and   | •  |                   |                    |
| Attached pleases find proof of profice perform. This proof of proficiency conciteria. These results have been significant of the proof of proficer. | onsists of 4 LCSs                          | run consecutively | that meet QC       |
| The employee listed above has been for which he/she has been trained.   | n approved to perf                         | form the analyses | s/responsibilities |
|   | -  | Supervisor/       | <br>Date           |

# APPENDIX V BENCH LEVEL DATA REVIEW CHECK LIST

#### **Bench Level Data Review Check List**

| Order ID   | Date     |    |     |         |
|--|----------|----|-----|---------|
| Test   | Batch ID |    |     |         |
| Have you checked the following items carefully before you reported the data? |          |    |     |         |
| Check Point  | Yes      | No | N/A | Comment |
| Is there a COC note?   |          |    |     |         |
| Did initial calibration pass, including blank?                               |          |    |     |         |
| Any positive readings in your Mblank?  |          |    |     |         |
| Did your CCV's Pass?   |          |    |     |         |
| Did you have analyte carry over?   |          |    |     |         |
| Did you check your D. F.?  |          |    |     |         |
| Is this report for TRRP?   |          |    |     |         |
| If TRRP, did you use dry weight basis?                                       |          |    |     |         |
| Is your analysis started within holding time?                                |          |    |     |         |
| Has your standard or buffer passed its expiration date?                      |          |    |     |         |
| Did your sample Dup RPD meet the criteria?                                   |          |    |     |         |
| Did your LCS & LCSD meet the criteria for % Rec.?                            |          |    |     |         |
| Did your MS & MSD meet the criteria for % Rec.?                              |          |    |     |         |
| Did your LCS & LCSD meet the criteria for RPD                                |          |    |     |         |
| Did your MS & MSD meet the criteria for RPD?                                 |          |    |     |         |
| Did you run a rep QC if needed?  |          |    |     |         |
| Are your sample readings bracketed by standards?                             |          |    |     |         |
| Special Comments:  |          |    |     |         |
| Analysts Signature:  |          |    |     |         |

Appendix VI Test Methods Used by A&B

## Summary of Test Methods used by A&B

| Method        | Description                        | Current Certification |
|---------------|------------------------------------|-----------------------|
| 3M 3500       | Organic by 3M Badge                | AIHA                  |
| 3M 3551       | Ethylene Oxide by 3M Badge 3551    | AIHA                  |
| ABL GC02      | BTEX in Air - Tedlar Bag           |                       |
| ABL IC01      | IC                                 |                       |
| ABL IC02      | Amines by IC                       |                       |
| ABL LC01      | Anions by HPLC                     |                       |
| AOAC 984.27   | Iron in Food Products by ICP       |                       |
| ASTM 808-81   | Chlorine                           |                       |
| ASTM D129-64  | Sulfur                             |                       |
| ASTM D-240    | BTU                                |                       |
| ASTM D482-95  | Ash                                |                       |
| CMMEF 20.54   | Yeasts and Molds                   |                       |
| CMMEF 20.61   | Yeasts and Molds                   |                       |
| CMMEF 35.14   | E. Coli 0157:H7                    |                       |
| CMMEF 35.24   | E. Coli 0157:H7                    |                       |
| CMMEF 36.53   | Listeria                           |                       |
| CMMEF 37.74   | Salmonella                         |                       |
| CMMEF 39.54   | Staphylococcus aureus C+           |                       |
| CmMEF 39.58   | Staphylococcus aureus C+           |                       |
| CMMEF 40.13   | Vibrio                             |                       |
| CMMEF 7.63    | Plate Count                        |                       |
| CMMEF 8.935   | Coliform - Petrifilm               |                       |
| D 1744        | Water by Karl Fischer              |                       |
| Enterotube    | Bacteria Speciation                |                       |
| EPA 110.2     | Color                              |                       |
| EPA 120.1     | Specific Conductance               |                       |
| EPA 150.1     | рН                                 | NELAP                 |
| EPA 160.1     | TDS                                | NELAP                 |
| EPA 160.2     | TSS                                |                       |
| EPA 160.3     | Total Solids                       | NELAP                 |
| EPA 160.4     | Volatile Solids                    |                       |
| EPA 1664      | Oil and Grease, Hexane Extractable |                       |
| EPA 180.1     | Turbidity                          |                       |
| EPA 200.7     | Metals by ICP                      | NELAP, TCEQ           |
| EPA 204.2     | Antimony by GFAA                   |                       |
| EPA 213.2     | Cadmium by GFAA                    |                       |
| EPA 218.4     | Hexavalent Chromium                |                       |
| EPA 239.2     | Lead by GFAA                       |                       |
| EPA 245.1     | Mercury                            | NELAP, TCEQ           |
| EPA 270.2     | Selenium by GF/AA                  |                       |
| EPA 300       | Anions by IC                       | NELAP, TCEQ           |
| EPA 300/200.7 | NaCl                               |                       |
| EPA 305.1     | Acidity                            |                       |
| EPA 310.1     | Alkalinity                         |                       |
| EPA 325.3     | Chloride                           |                       |

| Method           | Description                         | Current Certification |
|------------------|-------------------------------------|-----------------------|
| EPA 330.5        | Residual Chlorine                   |                       |
| EPA 335.2        | Total Cyanide                       |                       |
| EPA 340.2        | Fluoride by Ion Selective Electrode |                       |
| EPA 350.3        | Ammonia Nitrogen                    |                       |
| EPA 350.3/351.4  | Total Organic Nitrogen              |                       |
| EPA 351.4        | TKN                                 |                       |
| EPA 351.4/300    | Total Nitrogen                      |                       |
| EPA 360.1        | DO                                  |                       |
| EPA 365.2        | Phosphorus                          |                       |
| EPA 375.4        | Sulfate                             |                       |
| EPA 376.1        | Sulfide                             |                       |
| EPA 405.1        | BOD                                 |                       |
| EPA 410.4        | COD                                 |                       |
| EPA 413.1        | Oil and Grease, Freon Extracted     |                       |
| EPA 415.1        | TOC                                 |                       |
| EPA 418.1        | TPH, IR                             |                       |
| EPA 420.1        | Total Phenols                       |                       |
| EPA 445          | Chlorophyll a                       |                       |
| EPA 445.0        | Pheophytin a                        |                       |
| EPA 524.2        | Trihalomethanes (Total)             | TCEQ                  |
| EPA 552.2        | Haloacetic Acids (Total)            | TCEQ                  |
| EPA 602          | BTEX                                | TOLQ                  |
| EPA 608          | Organochloro Pesticides and PCBs    |                       |
| EPA 610          | PAH by HPLC                         |                       |
| EPA 615          | Chlorinated Herbicides              |                       |
| EPA 624          | VOC                                 |                       |
| EPA 625          | SVOC                                |                       |
| EFA 025          | 3,000                               |                       |
| EPA/600/R-93/116 | Asbestos by PLM                     |                       |
| FDA E7C23        | Bacteria Screen for Cosmetics       |                       |
| LA 29-B          | Louisiana 29-B                      |                       |
| LaMotte 3347     | Ferrous and Ferric Iron             |                       |
| NIOSH 1024       | IH 1,3 Butadiene                    |                       |
| NIOSH 1300       | IH Acetone and Styrene              |                       |
| NIOSH 1400       | IH t-Butanol                        |                       |
| NIOSH 1500       | IH Organics in tube                 |                       |
| NIOSH 1501       | ін тнс                              |                       |
| NIOSH 1552       | IH d-Limonene and Limonene          |                       |
| NIOSH 1603       | IH Acetic Acid                      |                       |
| NIOSH 2000       | IH Methanol                         |                       |
| NIOSH 2016       | IH Formaldehyde                     |                       |
| NIOSH 2540       | IH Diethylenetriamine               |                       |
| NIOSH 2546       | IH Cresols                          |                       |
| NIOSH 3500       | IH Formaldehyde                     |                       |
| NIOSH 500        | IH Dust                             |                       |
| NIOSH 5020       | IH Dibutyl Phthalate                |                       |
| NIOSH 5026       | IH Oil Mist                         |                       |

| Method          | Description                          | Current Certification |
|-----------------|--------------------------------------|-----------------------|
| NIOSH 5503      | IH PCB                               |                       |
| NIOSH 600       | IH Respirable Dust                   |                       |
| NIOSH 6007      | IH Nickel Carbonyl                   |                       |
| NIOSH 6009      | IH Mercury Vapor                     |                       |
| NIOSH 7300      | IH Metals - Cassette                 |                       |
| NIOSH 7600      | IH Chromic Acid                      |                       |
| NIOSH 7903      | IH Anions                            |                       |
| NIOSH 9100      | IH Metals Scan - Wipe                | AIHA                  |
| OSHA 5517 (M)   | IH 1,2,4-Trichlorobenzene            |                       |
| OSHA 67         | IH Pesticides                        |                       |
| OSHA 1550       | IH Naptha                            |                       |
| OSHA 42 M       | IH Isocyanates                       |                       |
| OSHA 56         | IH 1,3-Butadiene                     |                       |
| OSHA 64         | IH Glutaraldehyde                    |                       |
| Rad             | Radiation Screen                     |                       |
| S&B 010016      | Hemolytic                            |                       |
| S&B 110138      | Bacillus                             |                       |
| S&B 560466      | Lactic Acid Bacteria                 |                       |
| SKC 575         | THC - SKC Badge                      |                       |
| SM 2340B        | Hardness                             |                       |
| SM 2520 B       | Salinity                             |                       |
| SM 2540 G       | % Moisture                           |                       |
| SM 2540B        | % Water                              |                       |
| SM 2550B        | Temperature                          |                       |
| SM 2710 F       | Density                              |                       |
| SM 3500         | Hexavalent Chromium                  |                       |
| SM 4500         | Free CO2                             |                       |
| SM 4500CN-C,E,G |                                      | TCEQ                  |
| SM 4500CN-C,L,G | Weak Acid Dissociable Cyanide        | TOLQ                  |
| SM 4500B        | Sulfite or Chloride                  |                       |
| SM 5210         | CBOD                                 |                       |
|                 |                                      | TCEQ                  |
| SM 9215 B       | Heterotropic Plate Count             | TOEQ                  |
| SM 9215 C       | Heterotropic Plate Count             |                       |
| SM 9215 D       | Heterotropic Plate Count             |                       |
| SM 9222 C       | Total Coliform                       |                       |
| SM 9222 D       | Fecal Coliforms                      |                       |
| SM 9222B        | Total Coliforms                      |                       |
| SM 9222E        | E. coli                              | TOTO                  |
| SM 9223         | Total Coliform/E. coli               | TCEQ                  |
| SM 9230C        | Fecal Streptococcus and Enterococcus |                       |
| SM 9240D        | Sulfate Reducing Bacteria            |                       |
| SM 9260 J       | Legionella                           |                       |
| SM 9260D        | Salmonella                           |                       |
| SM 9260E        | Shigella                             |                       |
| SM 9260F        | Pathogenic E.Coli                    |                       |
| SM 9260G        | Campylobacter                        |                       |
| SM 9260H        | Vibrio                               |                       |

| Method       | Description                               | Current Certification |
|--------------|---|-----------------------|
| SM 9260K     | Yersinia                                  |                       |
| SW-846 1010  | Ignitability                              | NELAP                 |
| SW-846 1311  | TCLP Procedure                            | NELAP                 |
| SW-846 6010B | Metals                                    | NELAP                 |
| SW-846 7.3   | Reactive Cyanide/ Sulfide                 |                       |
| SW-846 7041  | Antimony by GFAA                          |                       |
| SW-846 7191  | Chromium                                  |                       |
| SW-846 7470A | Mercury                                   | NELAP                 |
| SW-846 7471A | Mercury in solids                         |                       |
| SW-846 7841  | Thallium by GFAA                          |                       |
| SW-846 8015B | GC/FID                                    |                       |
| SW-846 8021B | BTEX                                      | NELAP                 |
| SW-846 8081A | Organochlorine Pesticides                 | NELAP                 |
| SW-846 8082  | PCBs                                      |                       |
| SW-846 8151A | Chlorinated Herbicides                    | NELAP                 |
| SW-846 8260B | VOC                                       | NELAP                 |
| SW-846 8270C | SVOC                                      | NELAP                 |
| SW-846 8310  | PAH by HPLC                               |                       |
| SW-846 8315A | Formaldehyde by HPLC                      |                       |
| SW-846 8316  | Acrylamide/Acrylonitrile/Acrolein by HPLC |                       |
| SW-846 8330  | Nitroaromatics and Nitroamines by HPLC    |                       |
| SW-846 9010B | Cyanide                                   |                       |
| SW-846 9020B | TOX                                       |                       |
| SW-846 9030B | Sulfide                                   |                       |
| SW-846 9040C | рН  | NELAP                 |
| SW-846 9045  | рН  |                       |
| SW-846 9060  | TOC -                                     |                       |
| SW-846 9095A | Paint Filter                              |                       |
| SW-846 9212  | Chloride - by ISE                         |                       |
| TO-10        | Pesticides in Air                         |                       |
| TO-11A       | Formaldehyde in Air                       |                       |
| TO-15        | Air Analysis - Cannister                  | NELAP                 |
| TX 1005      | Total Petroleum Hydrocarbons              |                       |
| TX 1006      | Aliphatic and Aromatic Hydrocarbons       |                       |
| WWWTP Ch 10  | Microscopic Sludge Evaluation             |                       |